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PATENT COOPERATION TREATY

REC'D 30 AUG 2001	
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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant s or agent s file reference 10G	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
International application No. PCT/RU 00/00209	International filing date (day/month/year) 31 May 2000 (31.05.2000)	Priority date (day/month/year) 31 May 1999 (31.05.1999)
International Patent Classification (IPC) or national classification and IPC G01B 7/34, G01N 27/00		
Applicant GIVARGIZOV Evgeny Invievich et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This Report consists of a total of 3 sheets, including this cover sheet.

☐ This report is also accompanied by ANNEXES, i.e., sheets of the description, claimes and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under PCT).

These annexes consist of a total of ______ sheets

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Articl 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand: 29 December 2000 (29.12.2000)	Date of completion of this report: 05 August 2001 (05.08.2001)
Name and mailing address of the IPEA/ RU FIPS Russia, 121858, Moskva, Berezhkovskaya nab., 30-1 Facsimile No.	Authorized officer E.Andreichenko Telephone No 240-25-91

Form PCT/IPEA/409 (cover sheet)(July 1998)

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/RU 00/00209

I. Basis of the report

1. With regard to the **elements** of the international application:*

☒ the international application as originally filed

☐ the description:
pages __________, as originally filed
pages __________, filed with the demand
pages __________, filed with the letter of __________

☐ the claims:
pages __________, as originally filed
pages __________, as amended (together with statement) under Article 19
pages __________, filed with the demand
pages __________, filed with the letter of __________

☐ the drawings:
pages __________, as originally filed
pages __________, filed with the demand
pages __________, filed with the letter of __________

☐ the sequence listing part of the description:
pages __________, as originally filed
pages __________, filed with the demand
pages __________, filed with the letter of __________

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.
These elements were available or furnished to this Authority in the following language __________ which is:
☐ the language of a translation furnished for the purposes of international search (under Rule 23.1.(b)).
☐ the language of publication of the international application (under Rule 48.3(b)).
☐ the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:
☐ contained in the international application in written form.
☐ filed together with international application in computer readable form.
☐ furnished subsequently to this Authority in written form.
☐ furnished subsequently to this Authority in computer readable form.
☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. ☐ The amendments have resulted in the cancellation of:
☐ the description, pages __________
☐ the claims, Nos. __________
☐ the drawings, sheets/fig. __________

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).**

* *Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).*

** *Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.*

V. Reasoned statement under Article 35(2) with regard to n velty, inventive step or industrial applicabillity; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims	1 - 74	YES
	Claims		NO
Inventive Step (IS)	Claims	1 - 74	YES
	Claims		NO
Industrial Applicability (IA)	Claims	1 - 74	YES
	Claims		NO

2. Citations and explanations (Rule 70.7)

The examination report has been established on the basis of all the documents cited in Search report. The following documents are considered to be the closest prior art for the claimed inventions:

D1 - US 5367165 D3 - WO 97/37064

D2 - US 5742377 D4 - US 5811017

D1, D3, D4 disclose the tip structures, the methods for preparation of the tip structures and the cantilevers including the tip structures. D2 discloses the cantilever, the method for its preparation and the scanning probe device including the cantilever. But none of these documents discloses the features of claim 1 and 30, namely the axis of the tip forming a given angle in respect to the vertical that passes through its basis. None of these documents discloses the features of independent claims 15 and 46 concerning the tip not epitaxial to the substrate. None of the documents discloses the features of claim 51 concerning the tip implemented as a tip structure according to claims 1-29. The invention according to claim 67 differs from known by that a method for preparation of a cantilever includes a formation of at least one lever from the first conducting layer and a formation of at least one electrode arranged along the lever at a side opposite to the probe from the second conducting layer.

The claimed inventions provide the possibility of creating new designs of electron devices such as scanning probes and field emitters based on tip structures.

Consequently claims 1-74 are novel and involve an inventive step.

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
7 December 2000 (07.12.2000)

PCT

(10) International Publication Number
WO 00/74107 A3

(51) International Patent Classification[7]: G01B 7/34, G01N 27/00

(21) International Application Number: PCT/RU00/00209

(22) International Filing Date: 31 May 2000 (31.05.2000)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:

99111382	31 May 1999 (31.05.1999)	RU
99111432	31 May 1999 (31.05.1999)	RU
2000107025	23 March 2000 (23.03.2000)	RU
2000107026	23 March 2000 (23.03.2000)	RU

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(81) Designated States *(national)*: AT, AU, BG, BR, CA, CH, CN, CZ, DE, DK, ES, FI, GB, HU, IL, IN, JP, KR, LT, LU, PL, RU, SE, SG, SI, SK, UA, US.

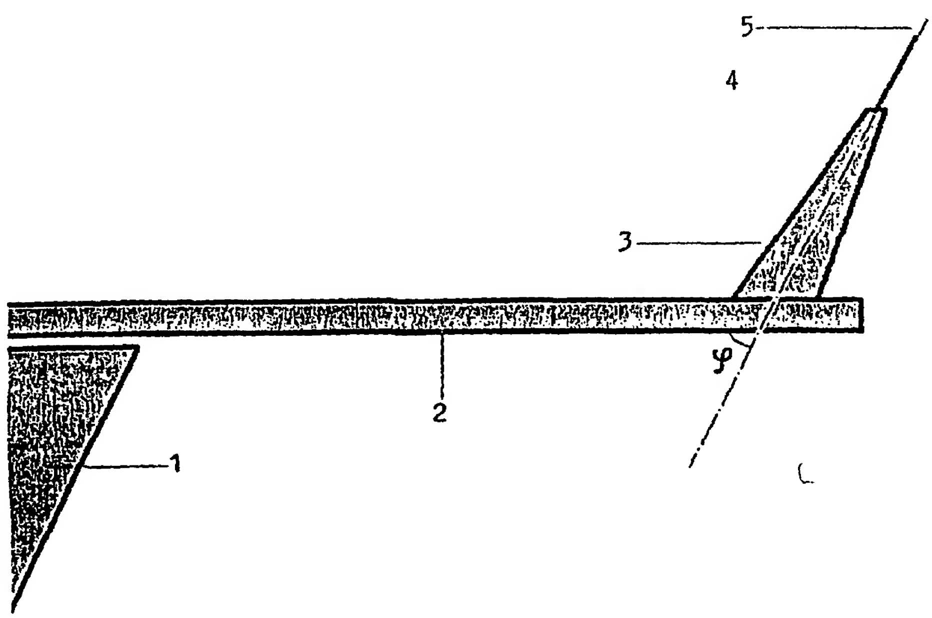
(84) Designated States *(regional)*: European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

Published:
— *With international search report.*

(88) Date of publication of the international search report: 22 March 2001

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: TIP STRUCTURES, DEVICES ON THEIR BASIS, AND METHODS FOR THEIR PREPARATION



(57) Abstract: New designs of electron devices such as scanning probes and field emitters based on tip structure are proposed. The tips are prepared from whiskers that are grown from the vapor phase by the vapo-liquid-solid technology. The tip structure includes a single crystalline substrate and a single crystalline tip. The axes of the tip forms a given angle in respect to the vertical that passes through its basis.

WO 00/74107 A3

INTERNATIONAL SEARCH REPORT

International Application No
PCT/RU 00/00209

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 G01B7/34 G01N27/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 G01B G01N G01R C30B H01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

PAJ, WPI Data, INSPEC, COMPENDEX, EPO-Internal, IBM-TDB

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 367 165 A (TODA AKITOSHI ET AL) 22 November 1994 (1994-11-22) claims 1-9	1,51,52
A	US 5 742 377 A (MINNE STEPHEN CHARLES ET AL) 21 April 1998 (1998-04-21) cited in the application claims 1-38	51,52,61
A	WO 97 37064 A (GIVARGIZOV EVGENY INVIEVICH) 9 October 1997 (1997-10-09) claim 1	30,46
A	US 5 811 017 A (MATSUYAMA KATSUHIRO) 22 September 1998 (1998-09-22) claim 1	1,30

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
29 November 2000	18/12/2000

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Van den Bulcke, E

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/RU 00/00209

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 021 364 A (AKAMINE SHINYA ET AL) 4 June 1991 (1991-06-04) claims 1-16	1,30
A	US 5 903 161 A (NAKANO TATSUO ET AL) 11 May 1999 (1999-05-11) claims 1,10,11	1
A	EP 0 566 214 A (CANON KK) 20 October 1993 (1993-10-20) claims 1-4	1
A	A.A.NOSOV ET AL.: "the influence of an electric field on the formation of gold whisker single crystals" RADIO ENGINEERING AND ELECTRONIC PHYSICS, vol. 17, no. 2, February 1972 (1972-02), pages 281-286, XP002154139 page 281 -page 286; figures 2-7	1
A	K.L.LEE ET AL.: "SUBMICRON Si TRENCH profiling with an electron -beam fabricated atomic force microscope tip" JOURNAL VACUUM SCIENCE TECHNOLOGY , vol. B9, no. 6, November 1991 (1991-11), pages 3562-3568, XP000965676 cited in the application page 3564; figure 3B	1
A	E.I.GIVARGIZOV: "ultrasharp tips for field emission applications prepared by the vapor-liquid-solid growth technique" JOURNAL VACUUM SCIENCE TECHNOLOGY, vol. b, no. 11(2), March 1993 (1993-03), pages 449-453, XP000965673 cited in the application page 449; figure 453	

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/RU 00/00209

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5367165 A	22-11-1994	JP 6052820 A	25-02-1994
		JP 5256643 A	05-10-1993
US 5742377 A	21-04-1998	US 5517280 A	14-05-1996
		US 5883705 A	16-03-1999
		US 6075585 A	13-06-2000
		US 5618760 A	08-04-1997
		US 6000947 A	14-12-1999
		US 5666190 A	09-09-1997
WO 9737064 A	09-10-1997	RU 2099808 C	20-12-1997
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US 5811017 A	22-09-1998	JP 8313541 A	29-11-1996
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EP 0566214 A	20-10-1993	JP 2069618 A	08-03-1990
		JP 2071439 A	12-03-1990
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		JP 2553661 B	13-11-1996

TIP STRUCTURES, DEVICES ON THEIR BASIS, AND METHODS FOR THEIR PREPARATION

FIELD OF THE INVENTION

The invention relates to materials sciences, especially to electronic materials sciences, in particular to microelectronics, especially to emission electronics, to precision instruments for scientific and technological research. More specifically, this invention relates to design and to methods for preparation of electron emissive devices and of scanning probe devices. This invention can also be used in lithographic processes, as well as in basic processes of micro- and nanoelectronics.

PRIOR ART

Electron emissive devices (EED) represent the devices giving flows of electrons in the vacuum for various aims: for optical imaging, for electron-beam lithography, for lighting, etc. A cathode (an emitter) bearing the flows of electrons represents a principal component of the devices. Thermal cathodes heated to high temperatures serve as a classic example of such devices. However, the thermal cathodes consume a lot of energy for their operation. In this respect, field emission cathodes (or "cold cathodes") are far more effective devices. So-called Spindt cathodes based on molybdenum tips could serve as an example of the field emission devices [1]. Devices based on semiconductor (silicon) field emitters [2] are more suitable for applications due to cheapness of the materials and their technology.

Field emission devices based on silicon tips prepared from silicon whiskers (filamentary crystals) are known [3]. In particular, an idea of using of the resistance of the silicon emitter itself as a ballast resistance, that is important for field-emission displays (FED), has been realized in the patent [3]. In addition, the emitter was coated by diamond for increasing of the emission ability and of its durability [3].

This invention allows to increase the efficiency of the emission owing to the increase of number of emitters having the same spatial coordinate. Accordingly, a given pixcell can increase the emissivity brightness several times.

Carbon nanotubes on flat substrates used in the field emitters are known [4]. However, parameters of such emitters are not reproducible because distributions of electric fields between the nanotubes is non-uniform due to their occasional positions.

Scanning probe microscopes (SPM) are able to give images of solid surfaces with high spatial resolutions. A use of carbon nanotubes attached to the probes is known [5]. However, their position at the probe is non-controllable due to their occasional and numerous nucleations.

The SPM can be used for study of magnetic objects with a high resolution and high sensitivity. Probe tips for the instruments are made of a non-magnetic material (such as silicon) coated by a film of magnetic material (such as iron, cobalt, etc) [6-8]. However, both a shape and a structure of the coatings used in the papers are non-optimal for the high resolution and the high sensitivity of the instruments.

The SPM for electrical capacitance measurements uses probes that made of silicon tips [9, 10]. However, both a shape of the tips and a composition of the capacitance material are not optimal for high sensitivity of the instrument.

SPM probes with side tips for profile studies are known [11]. However, the probes are suitable only for studies of surfaces having rather simple forms such as grooves with vertical walls. Meantime, there are a lot of cases where surfaces with complicated shapes (such as biological macromolecules) or with a coarse relief must be studied.

There are problems with mapping the spatial arrangement of chemical forces existing on solid surfaces [12].

Problems with ensuring high scanning rates in SPM devices having a single lever/probe are known. Due to the small scanning rate, such devices are not still broadly used in industry.

A multi-lever device has been proposed in [13]. In the device, a signal from each probe is treated in a microchip that is placed on a holder. After treating the signal, it is applied to a system for controlling a variety of levers. In this operation, piezoresistive layers are used. Drawbacks of the multi-lever device are the following:

1.In order to realize both feeding/taking-off the levers and tracing their deflections, the using only the piezoresistive layers is not sufficient.

2.Creation/production of the multilever devices integrated with multiplexers, operational amplifiers, etc (that is necessary for an effective action of the multi-lever devices) represents a very complicated and expensive technological problem.

A cantilever for a SPM, as well as techniques for a registration and for treatments of signals obtained, are proposed in patent [14]. In particular, a device is proposed that is based on a measurement of change of the capacity between the lever and neighbouring stationary plane. The device includes also a controlled action to the lever by an electrostatic interaction between the stationary plane and the lever (Fig.). In this case, three principal tasks are solved:

- application of resonance modes to the lever when it acts in the taping mode;
- electrostatic feeding/taking-off the lever;
- control of the lever deflection by the measurement of the capacity.

However, sometimes, especially at the action of the SPM in the regime of the point scanning of adhesion forces [15], an ability of the device to ensure a fast damping of non-resonant oscillations, to damp the lever for its subsequent interaction with solid surface under study is very important. Such a property of the device, as well as a suitable design of the cantilever, can substantially (3-5 times) decrease the time of investigation of the surface.

In order to realize such a property, it is proposed in this invention to use an actuator – a part of the cantilever that is rigidly connected with its holder. When the probe is detached off the surface (where it was, e.g., "captured" by the adhesion forces), non-resonant oscillations of the lever arise. By measurements of changes of the capacity, existing between the lever and the actuator, the oscillations are applied to the input of the system that has a negative feed-back: a similar (by amplitude) and an opposite (in sign) signal is applied to the actuator. This results in the non-resonant damping of the lever oscillations and, finally, in its relaxation.

Thus, in this invention, in addition to the approaches developed in the patent [14], we propose an approach that ensures a stable and fast action of the scanning probe device in any regimes of its work.

To this aim, we propose to provide the cantilever with a second electrode that applies the resonant modes of oscillations to the lever.

In this invention, a rational design of the multi-lever and a non-expensive technology for its production is proposed.

SUMMARY OF THE INVENTION

A uniqueness of the present invention consists in the following. Stages of preparation of important components of scanning probe devices (SPD) such as levers, probes on them, etc, can be fractionized and separated each of other by using a new technology for preparation of tip probes. Owing to this fact, it is possible to form multifunctional tip structures that allows to combine in a given device, a multilever, several probes with various sensitive components for simultaneous implementation of morphological, electrostatic, magnetic, chemical, etc, investigations.

A tip structure that includes a single-crystalline substrate and a single-crystalline tip epitaxial to the substrate, so that the axis of the tip forms a given angle in respect to the vertical that passes through its basis, the substrate could has a plane surface or represents a single-crystalline tip epitaxial to a plane single-crystalline surface. A single point of the substrate can serves as a basis for at least two tips. The tip can has a shape that includes at least one step and two links, the axis of each subsequent link can form its own angle in respect to the axis of the previous link. Also at least one of the steps serves as a basis for at least two links, at least one of them can be not epitaxial to the previous one. In principal it is possible that at least one of the links is formed by a nanotube, beeing so the nanotube can be combined by layers of different materials, one of them being carbon and at least one of the links can be formed by at least one atomic row. Also at least one tip can has a particle on its top that contains, in addition to the tip material, at least one more chemical element, the particle can be coated by a film of this or another element, and at least one chemical element, that is contained in the particle, can participates in the growing of the tip structure, and the particle can be coated by a film of this or another element. For special tasks chemical functional groups are deposited in the film. This invention proposes that a non-magnetic tip has a flat top, a monodomenic magnetic particle of a conical shape is placed on the flat top, basis of the particle contacting with the flat top. Also an electroconductive tip has a flat top perpendicular to the axis of the tip, the flat top is coated by a dielectric film, a p-n junction in the upper part of the tip being parallel and close to the flat top.

According to this invention a tip structure that includes a substrate and a single-crystalline tip can be so that the tip is not epitaxial to the substrate. Also axis of the tip forms a given angle in respect to the vertical that passes through its basis, the

substrate could has a plane surface or represents a single-crystalline tip epitaxial to a plane single-crystalline surface. A single point of the substrate can serves as a basis for at least two tips. The tip can has a shape that includes at least one step and two links, the axis of each subsequent link can form its own angle in respect to the axis of the previous link. Also at least one of the steps serves as a basis for at least two links, at least one of them can be not epitaxial to the previous one. In principal it is possible that at least one of the links is formed by a nanotube, beeing so the nanotube can be combined by layers of different materials, one of them being carbon and at least one of the links can be formed by at least one atomic row. Also at least one tip can has a particle on its top that contains, in addition to the tip material, at least one more chemical element, the particle can be coated by a film of this or another element, and at least one chemical element, that is contained in the particle, can participates in the growing of the tip structure, and the particle can be coated by a film of this or another element. For special tasks chemical functional groups are deposited in the film. This invention proposes that a non-magnetic tip has a flat top, a monodomenic magnetic particle of a conical shape is placed on the flat top, basis of the particle contacting with the flat top. Also an electroconductive tip has a flat top perpendicular to the axis of the tip, the flat top is coated by a dielectric film, a p-n junction in the upper part of the tip being parallel and close to the flat top.

This invention propose a method for preparation of the tip structure by means of epitaxial growing of the tip according to the vapor-liquid-solid mechanism on a substrate by deposition from a vapor-gaseous and/or gaseous mixture with using of at least one metallic solvent, so being the tip structure is growing as at least one tip so that the axis of the tip forms a given angle in respect to the vertical that passes through its basis. As the substrate a single-crystalline wafer oriented along a certain crystallographic plane can be used, this single-crystalline wafer allows to prepare the tip structure as at least one tip epitaxial to the substrate under an angle to its surface or a single-crystalline tip epitaxial to a flat single-crystalline surface can be used as the substrate. Also the tip structure which are mentioned above can be prepared by a changing the growing temperature and/or concentrations of compounds in the vapor-gaseous or gaseous mixture, and/or pressures of the vapor-gaseous or gaseous mixture, and/or by addition of at least one metallic solvent and/or its evaporation. Also after the growing the tip structure a diffusion of at least one chemical element into the structure is performed with conservation of the structure of at least one

metallic solvent. After the preparation of the structure it can be immersed into an amorphous material, the composite obtained can be polished together with at least one apex of the tip structure until formation of a flat surface, and the amorphous material can be etched away. After the etching away the amorphous material, a diffusion of at least one chemical element into the material of the tip structure can be performed. A diffusion of at least one chemical element into at least one metallic solvent can be performed. Then by etching off the material that has diffused into at least one metallic solvent, the metallic solvent can be removed. And at least one chemical element can be evaporated onto all the surface of the tip structure. A part of the evaporated chemical element can be removed by an etching off the diffusion layer from the surface of the tip structure, or by etching off the amorphous layer with conservation of the chemical element on at least one apex. The epitaxial growing of the tip structure can be implemented, and by a changing the growing temperature and/or concentrations of compounds in the vapor-gaseous or gaseous mixture, and/or pressures of the vapor-gaseous or gaseous mixture, and/or by addition of at least one metallic solvent and/or its evaporation a step and/or a plateau on at least one apex can be created, after that the solidified globule can be removed. And at least one of the procedures described above can be used at least one more time. This invention also propose methode for preparation of the tip structure according which at least one time a magnetic material is used as the evaporating one, the magnetic particle formed is sharpened by a bombardment with accelerated ions, and a monodomenization of the particle can be performed. So being the monodomenization can be performed by enduring of the particle in a constant magnetic field of a certain direction. And the monodomenization can be performed at a high temperature of the magnetic particle, the temperature can be reached by passing a field-emission current through the tip structure.

This invention also propose a method for preparation of at least one tip structure by directional growing according to the vapor-liquid-solid mechanism on a substrate at deposition from a vapor-gaseous and/or gaseous mixture with using of at least one metallic solvent, so that the tip structure is grown non-epitaxially to the substrate. And the tip structure can be created according to the mention above points.

Also hollow can be created in the substrate for the growing of the tip, andthe hollow can has a shape that corresponds to the crystallographic structure of the tip material.

According to this invention a source of electrons that includes a substrate, a field emitter, and a source of charge carriers, so being the field emitter represents a tip structure according to the mentioned above points.

According to this invention a cantilever for scanning probe devices that includes a holder, a lever and a probe, so being the tip is implemented as a tip structure according to the mentioned above points.

According to this invention a cantilever for scanning probe devices that includes: at least two alternating plane-parallel layers of conducting materials separated by non-conducting layers; at least one bending section – a lever implemented from a first conducting layer;a probe placed on the lever; at least one electrode – a section of a second conducting layer arranged along the lever at the side opposite to the probe, so being the electrode contains a means for suppression of non-resonant oscillations of the lever, the means representing a feedback system. Also the probe can be implemented as a tip structure according to mentioned above points. And the electrode can contains a means for controlling the lever deflections, and/or a means for a forced deflection of the lever of its initial position, and/or a means for modulation of the resonant lever oscillations. Being so at the side of the lever that is opposite to the electrode side of the lever can be arranged another electrode implemented from an additional conducting layer and contained a means for controlling lever deflections, and/or a means for a forced lever deflection of its initial position, and/or a means for modulations of resonant oscillations of the lever, and/or a means for suppression of non-resonant oscillations of the lever, the system being acted as a feedback one. Also between the lever and at least one electrode a vacuum gap can exists, the gap can be filled by liquid and/or plastic material that allows a mutual shifting of the lever and the electrode relative to each other. Also the lever can has a П- and/or V-shape and/or longitudinal cavity, the cavity forming lever arms. Also the lever can has a piezoresistive layer and/or semiconductor layer doped up to the p^{++}-conductivity. Also the lever arms separated by a longitudinal section can has doped layers of n-, n^{+}-, p-, p^{+} type conductivity. And one of the lever arm can serves as a drain, another lever arm serves as a source for a control system, the arms being separated by a lever section that has another conductivity, one of the electrodes implements a function of a gate being a means of a control.

This invention also propose a scanning probe device that includes: a cantilever containing at least one lever, at least one controlling electrode and/or at least one

electrode for controlling lever deflections of an initial position; a system for regulation and controlling the lever deflections, being so the lever is implemented according to any of the mentioned above points. Also one electrode can be placed along at least two levers. And the system for controlling the lever deflections can represents a system for registration of the changes of the capacity between the lever and at least one electrode and/or of the contour quality, that includes its capacity, for each lever being chosen its own control frequency. Also the control system can includes a system for the forced deflection that can be electrostatic and/or electromagnetic one. According to the previous positions at least two electrodes can be placed along the same lever, and the system for controlling the lever deflections can represents a system for registration of the changes of the capacity between the arms of the lever, separated by a longitudinal section, and at least one electrode, for a controlling the lever rotation relative to a longitudinal axis a specific frequency being chosen for each of the lever arms.

This invention also propose a method for preparation of a cantilever for scanning probe devices that includes a formation of a composite wafer consisting of at least two alternating plane-parallel layers of conducting materials separated by non-conducting layers; a formation of at least one lever from the first conducting layer; a creation of a probe on the lever, so being that at least one electrode arranged along the lever at a side opposite to the probe is formed from the second conducting layer. And the probe can be implemented as the tip structure according to the mentioned above points. Also the composite wafer can be prepared by bonding of wafers and/or mechanical and/or chemical removal of parts of the wafers with conservation of thin layers having a given thickness. Also at least one conducting layer and/or at least one non-conducting layer of the composite wafer can be prepared by a deposition of a material and/or materials. And an electrode with contact outputs/terminals and/or a mechanico-electric structure for systems of control and/or regulation can be formed on at least one conducting layer before the bonding and/or between bonding stages and/or after the bonding. Being so an electrode with contact outputs/terminals and/or a mechanico-electric structure for systems of control and/or regulation can be formed on at least one conducting layer before the deposition and/or between the deposition stages and/or after the deposition of the material and/or materials. And at least one conducting layer and/or at least one non-conducting layer can be used at the

preparation of the cantilever as technological stop-layers. Also the probe can be implemented as the tip structure according to the mentioned above points.

A BRIEF DESCRIPTION OF THE FIGURES

Fig. 1. An array of silicon whiskers grown on a silicon substrate (111).

Fig. 2. A scheme of transformation of a silicon whisker, having a solidified globule at its apex, into a silicon tip:

a – an initial stage; b – an intermediate stage; c – a final stage;

1 – silicon substrate; 2 – body of the silicon whisker; 3 – the solidified globule consisting of fine crystallites of silicon and gold; 4 – the tip formed.

Fig. 3. A high-resolution-transmission-electron micrograph of the silicon tip.

Fig. 4. An array of silicon tips prepared from the array of silicon whiskers.

Fig. 5. A scheme (a) and a scanning-electron-micrograph (b) of a step-shaped silicon tip.

Fig. 6. An array of silicon columns, with flat tops, formed from the array of silicon whiskers.

Fig. 7. A scheme of the step-shaped silicon tip with a small plateau on its apex;

1 – silicon substrate (111); 2 – basic silicon whisker; 3 – silicon pin; 4 – top plateau.

Fig. 8. A scheme of a cathode tip structure with multiple-multiple-multiple (M3) field emitters formed by carbon nanotubes on silicon whisker tips;

1 – silicon substrate oriented along the plane (111); 2 – primary silicon whiskers = first link; 3 – secondary silicon whiskers = second link; 4 – nanotubes = third link; 5 – "step" = silicon whisker branching point; 6 – spatial coordinate.

Fig. 9. A scheme of the SPM probe whose sensitive tip is formed by a carbon nanotube;

1 – holder; 2 – lever; 3 – a silicon whisker basis; 4 – a silicon whisker pin; 5 – a nanotube; 6 – "dangling" chemical bondings; 7 – a single-atom row.

Fig. 10. A scheme of the SPM probe with an inclined silicon whisker tip;

1 – holder; 2 – silicon lever oriented along the silicon plane (111); 3 – silicon whisker basis; 4 – silicon whisker pin; 5 – nanotube.

Fig. 11. A scheme of a silicon tip coated by a magnetic particle.

1 – silicon substrate (111); 2 – basic silicon whisker; 3 – silicon pin; 4 – evaporated magnetic particle.

Fig. 12. A scheme of a silicon tip coated by a sharpened magnetic particle.

1- silicon substrate (111); 2- basic silicon whisker; 3 – silicon pin; 4 – sharpened magnetic particle.

Fig. 13. Prior art of the capacitance probes.

a – from [9]: a scheme of the probe, and a scheme of the measurements;

1 – probe, 2 – insulator (oxide), 3 – impurity;

b – from [10]: a scheme of the probe, and a scheme of the measurements;

1 - probe, 2 – oxide.

Fig. 14. A silicon tip with a flat apex coated by a dielectric.

1 – basic silicon whisker; 2 – silicon pin; 3 – dielectric film.

Fig. 15. A scheme of a SPM probe intended for investigations of samples having a complicated surface relief:

1 – holder; 2 – a basic silicon whisker grown in the direction <111>; 3 - a silicon whisker that continues epitaxially the basis one in the direction <111>; 4 – lever – (111) single crystalline silicon.

Fig. 16. Scanning electron micrograph of the step-shaped silicon tip/probe with the globule on its apex; the globule is shown by arrow.

Fig. 17. A scheme of the globule formed on the apex of the silicon whisker.

a – the globule is formed by a mixture of silicon and gold crystallites;

1 – whisker; 2 - silicon crystallite; 3 – gold crystallite;

b - the globule is formed by a mixture of crystallites of silicon, gold, and a third chemical element; 1 – whisker; 2 – silicon crystallite; 3 – gold

crystallite; 4 – crystallite of the third chemical element.

Fig. 18. A scheme of multi-lever device proposed in [13] .

1- micro wires; 2- probes; 3- multiplexor ; 4- operation amplifier.

Fig. 19. A scheme of multi-lever device proposed in [14] .

80- cantilever; 81- knife edge; 82- platform; 83- conductive region; 84- conductive region; 85- piezoresistor.

Fig. 20 a, b, c, d. Process of mono-crystalline growth of whisker not epitaxial to substrate.

a – metal solvent; b – first stage of growth; c – leveling of growth; d – final of growth;

1- hollow; 2- metal solvent; 3- parasites; 4- substrate surface; 5- whisker; 6- substrate.

Fig. 21 Cantilever with deflections indicator represented by electrode placed along lever.

1 – holder; 2 – silicon lever oriented along the silicon plane (111); 3 – silicon whisker basis; 4 – silicon whisker pin; 5 – nanotube; 6 – plastic material; 7 – electrode.

or of the

Fig. 22 Cantilever with integral system of control

1 – whisker probe; 2 – electrode of suppression of non-resonance, of deflections indicator and forced deflection system; 3 – silicon lever oriented along the silicon plane (111); 4 – electrode for modulation of the resonant lever oscillations;

Fig. 23 a, b, c. Multilever for scanning probe devices

1 - electrodes of suppression of non-resonance, of deflections indicator and forced deflection system; 2 – silicon lever oriented along the silicon plane (111); 3 - electrode for modulation of the resonant lever oscillations; 4, 5 – non-conducting layers; 6 – probe.

BEST VERSION FOR THE REALIZATION OF THE INVENTION

This invention is based on silicon tip probes prepared from silicon filamentary crystals ("whiskers") grown from the vapor phase according to the vapor-liquid-solid (VLS) process.

The growing process is performed as follows.

An array of gold dots, 5-7 μm in diameter, 0.2 μm in thickness, distanced each of other at 30 μm, is deposited onto a silicon wafer oriented along the most-close-packed crystallographic plane of silicon (111). The wafer is installed in the quartz chemical reactor and is heated to 800-900°C in a flow of the gaseous reaction mixture $SiCl_4$+H_2. The gold dots contacted to the silicon wafer form liquid droplets forming a solution of silicon in molten gold. The liquid droplets act as catalytic particles for the chemical reaction so that at the temperatures indicated it proceeds preferentially on the surface of the droplets. The liquid solution of silicon in gold formed becomes supersaturated, and the excess of the silicon is deposited at the droplet-substrate interface. As a result, an epitaxial filamentary crystal ("whisker") grows under each of the droplets.

The regular array of the whiskers is shown in Fig. 1. A solidified droplet ("globule") is seen at the apex of the each whisker. The globule consists of fine crystallites of silicon and gold.

Next, the whiskers are transformed into silicon tips by an chemical etching in a solution that act at silicon slowly. The etching is performed up to a stage when the globule is dropped down. The procedure is illustrated in Fig. 2.

A high-resolution micrograph of a tip is shown in Fig. 3.

A result of the transformation for the array of the whiskers is shown in Fig. 4.

If the whisker growing process is performed in two stages with different growth conditions (different temperatures, different concentration of $SiCl_4$ in the reaction mixture, etc), whiskers with a step shape can be formed. By the etching procedure illustrated in Fig. 2 a step-shaped silicon tip shown in Fig. 5 can be formed.

The array of the silicon whiskers can be transformed into an array of silicon "columns" (whiskers with flat plateau at their tops) if act to the globule, rather than to the body of the silicon whisker itself, for example, by etching the metallic solvent (gold), etc, see Fig. 6.

By combining the samples/procedures illustrated in Fig. 5 and Fig. 6, a step-shaped silicon tip with a small flat plateau at its apex can be formed, see. Fig. 7.

The tips and the arrays of the tips shown in Figs. 1 to 7 are used, according to this invention, for preparation of various devices.

One of the most typical, broad and important examples of the applications of the tip structures proposed in this invention is in the field-emission electronics.

Last years, large successes have been achieved in the field emission from carbon nanotubes. However, efficiency of the nanotube field emitters is strongly decreased when they are too dense so that the electric fields at their apices are highly lowered. It is known that the field emitters act independently each of other if they spaced at distances comparable with their heights.

An optimum design of the field emitters based on the nanotubes could be realized if combine regular silicon tip arrays with a branching of the whiskers and with formation of "bundles" of the nanotubes on their apices this is "multiple-multiple-multiple" (M^3) tip structure shown in Fig. 8. By choosing the distance between the regular silicon basic ("primary") whiskers, it is possible to increase multiply the field-emission currents from such tip structures.

An important improvement in the field emitters can be realized if provide simultaneous emission of a variety of the emitters. It is possible if different emitters have a sufficiently high resistance. In this case, the resistance can be considered as a "ballast resistance" in the electric circuit. Such an idea, patented in [3], can be realized in the M^3 tip structure (see Fig. 8b).

The same idea can be realized in the tip structure shown in Fig. 8a (M^2 tip structure) if a high "macroscopic" resistor incorporate in series to the tip structure.

The preparation process for carbon nanotubes can be combined with the VLS process that is used for whisker growing. This combination consists in the fact that a metal solvent used for whisker crystal growing can be used also as a catalyst for formation of the nanotubes.

SPM probes are other applications of the nanotube-on-silicon tip structures.

A scheme of such probes is shown in Fig. 9. Here, the small size of the apex area of the step-shaped silicon tips shown in Fig. 5 is illustrated as the silicon pin 4. It has an advantage because the probability of nucleation of the nanotubes depends on the apex area of the tips, and the smaller the area the larger is a chance to have there a

single nanotube necessary for the SPM probe. At an extreme case. the small size of the nanotube can be minimized down to a single-atom row 7.

Other versions of the SPM probes can use the step-shaped silicon tips shown in Figs. 5 and 7.

In particular, the SPM probe shown in Fig. 5b is suitable for applications in the semiconductor technology, e.g., for studying of the profiles of sub-micron-width grooves [16]. The nanometric ultra-sharp tip ensures high resolving power, whereas the relatively thick basis of such a probe ensures a sufficient mechanical stability against vibrations. For such cases, whisker probes are especially suitable [11, 17].

Another example for applications of the tip structure proposed in this invention is a special SPM probe for studies even more narrow (width less than 0.3 μm) and relatively deep (deepness more than 4-5 μm) grooves. This is a typical problem in the current and nearest-future semiconductor technology. The problem becomes especially difficult if the grooves are not vertical. To this aim, a SPM probe shown in Fig. 10 is suitable. The cantilever for such a probe is prepared from a composite plate that consists of a silicon wafer coated by a film of SiO_2 and a silicon layer having an orientation that is declined off the close-packed orientation (111) for angles φ about 15 to 25°. Such a relatively small angle allows to use a traditional technology for growing silicon whiskers according to the VLS mechanism shown above (see Fig. 1) and described in more detail in [18, 19]. The ability of silicon to grow in the direction [111] allows to prepare such probes.

The step-shaped silicon tip with the small plateau on its apex shown in Fig. 7 is suitable for preparation of the probes for the magnetic force microscopy (MFM) and for the electrical capacitance microscopy (ECM).

Typical size of the plateau is 100 nanometers in diameter. A magnetic (Fe, Co, or Ni) film about 100 nanometers in thickness is deposited onto the plateau by vacuum evaporation (Fig. 11). Calculation has shown that the resolving power of the MFM probe is better than 90 nanometers.

The magnetic particle having the cylindrical shape can be transformed into conical one by ion bombardment.

Example 1

A beam of argon or nitrogen ions accelerated by electrostatic field 5 kV is directed along the axis of the probe. A conical particle with an angle 40-50° at its apex

is formed, as it is shown in Fig. 12. The conical magnetic particle ensures the resolving power of the MFM about 50 nanometers. Calculation has shown that, at such a design of the probe, a contribution of the magnetic film deposited on the basis of the probe into a signal registered by the SPM is at least 100 times less than that of the conical particle deposited onto the tip.

The magnetic particle prepared by evaporation and, then, treated by the ion beam bombardment has a polycrystalline structure, each magnetic domen formed in a given crystalline grain having its own (arbitrary) direction of magnetization. In order to improve (homogenize) the magnetic properties of the particle, it is subjected to monodomenization by enduring the magnetic probe at a constant magnetic field of a given direction.

Examples of the prior art for the ECM probes are shown in Fig. 13. There, the tips of the probes have a hemispherical shape. Electrical capacity, the measurable parameter, depends on the shape and curvature radius of the tip and can be changed from tip to tip. In addition, the shape of the tip can be changed during the measurement procedure due to its possible contact with the solid surface studied.

In this invention, it is proposed to use in the ECM technique the step-shaped silicon tip with a flat plateau on its apex shown in Fig. 7. The tip is coated by a dielectric film such as Ta_2O_5 (having the value of dielectric constant 25 units, compare with 5 units for SiO_2), TiO_2 (100 units), $SrTiO_3$ (250 units), $BaTiO_3$ (1500 units) that have a high value of the dielectric constant (Fig. 14). This allows to work without contacting the tip with the surface studied. In addition, the flat shape of the tip surface facilitates exact calculations of the capacity and other related parameters.

Example 2

A thin film of titanium is deposited onto the silicon tip having the plateau. Then, the film is oxidized by heating to a high temperature in an oxygen-containing atmosphere. A TiO_2 coating is formed on the plateau.

Other problems that can be solved with the whisker based SPM probes relate to investigations of objects with complicated shapes (such as biological macromolecules, solid surfaces with a course relief, with arbitrary microcavities, etc). For investigations of such objects, a whisker probe having a shape shown in Fig. 15 can be used. The probe consists of at least two parts, a lower one and an upper one. The lower part is formed by a relatively thick silicon whisker perpendicular to a single-crystalline silicon substrate oriented along the most-close-packed

crystallographic plane (111) so that it has the orientation $<111>$. The upper part is formed by another, a more narrow whisker that grows in another, also crystallographic direction $<\bar{1}11>$ that forms the crystallographic angle $70^{\circ}32$ with the axis of the lower part.

One more version of the whisker based SPM probes relates to investigations of chemical constituents of matters, for example mapping the spatial arrangement of chemical forces existing on solid surfaces. The technique is named "chemical force microscopy" [12] and is based on discrimination of the chemical forces by specific material of the probe tip or by specific coatings the tip. Existing SPM probes made of silicon or silicon nitride have a weak adhesion ability in respect to the tip materials or tip coatings.

It is proposed in this invention to use silicon whisker probes whose tips are formed by the globules that contain, in addition to silicon, at least one more chemical element.

Such a probe is shown in Fig. 16. The globule contains, in addition to silicon, also a solvent (gold in this case) that participates in the whisker growth. Other materials (metals) can be added to the solvent so that the globule contains at least three chemical elements, see Fig. 17.

The globule can be coated by a thin film of the constituents having a good adhesion to it.

Example 3

Silicon whisker is grown by means of a mixture of gold and indium. Accordingly, the solidified globule contains, in addition to silicon, also crystallites of gold and indium. The whisker probe for the chemical force microscopy is coated with an indium film. Owing to the fact that some of indium crystallites of the globule are exposed at its surface, the adhesion of the indium film is enhanced.

Also, the globule, with the constituent coating or without it, can be coated with various chemical functional groups.

Example 4

A possibility to grow filamentary crystals - whiskers by the VLS process represents an important achievement of micro- and nano-electronics. The process allows to implement epitaxial growing the whiskers and to control the process. However, the necessity to use a substrate that has a certain crystallographic orientation is a drawback of the process. In this invention, it is proposed an approach

how to solve this issue. To this aim, in a substrate with an arbitrary (e.g., amorphous) structure a deep hollow ("well") is created, and a metallic solvent is placed on its bottom (Fig. 20). Such a substrate is installed in the chemical reactor for whisker growing. During the process crystalline nuclei are formed in the solvent droplet. At an initial stage, all the nuclei, except one nucleated at or close to the apex of the droplet, are at approximately equal conditions. The apex nucleus has a most abundant feeding. In addition, during the whisker growing, growth conditions for whiskers that have non-vertical component are deteriorated, accordingly, whiskers grow preferentially along the well.

In addition, the selective growth direction can be improved if the well has faceting in accordance with the direction of the preferential growth of a given material. For example, for silicon whiskers that are crystallizing in the diamond lattice the well should have a shape of equilateral triangles or hexagons.

Example 5

According to this invention, one of the version for tracking in the SPM system is realized in the cantilever design proposed in Fig. 21. Using silicon-on-insulator composite wafer (silicon (100) basic wafer/SiO_2 separating layer/silicon (111) film/SiO_2 separating layer/silicon (100) film) lever (111) is formed, a silicon whisker is grown on the lever, and a tip probe formed from the whisker. A plane electrode parallel to the lever is formed from the silicon film (100). The gap between the lever and the electrode is filled by a plastic material 6 (see Fig. 21). The filling the gap by the material allows electromechanical properties of such a system to keep constant.

By feeding resonant oscillations to this system an initial regime is specified that corresponds to a certain value of the quality factor of the resonant system. The quality factor is changed at the interaction of the probe with the surface studied. The change serves as the parameter that allows to track shifting the lever and, in such a way, to form an image of the surface studied.

Example 6

A most vivid example of implementation of the cantilever with integrated controlling systems for indication of deflections, for forced feeding/removal the lever relative to the surface studied, and for feeding the resonant oscillations to realize a "taping mode" regime is shown in Fig. 22. The electrode 4 placed along a small starting part of the lever has a gap in respect to the lever that is filled by a plastic material similarly to the Example 5. By feeding to the electrode 4 an alternating

voltage with a frequency of the electric field E_1 about 100 kHz between the lever 3 and the electrode 4, it is possible to excite resonant modulations of the lever without a necessity to feed a mechanical oscillation to the holder of the probe (that represents a significant hindrance at researches in liquids when mechanical oscillations excite strong secondary – non-desirable! – modes of oscillations in the liquid medium).

In the example considered, the electrode 2 contains three means for controlling the lever behavior: an electrostatic system E_2 for forced deflections, a system E_3 for indication of deviations by measurement of capacity between the lever 3 and the electrode 2, and a system E_4 for suppression of non-resonant oscillations (e.g., secondary oscillations appearing at discontacting the probe off the surface, the contact originating by adhesion forces). The last one (E_4) operates according to the following principle. A constant electrostatic charge is applied to the lever. When non-resonant oscillations appear at the lever, a corresponding charge is induced at the electrode 2 with the frequency of the above (initial) oscillations. The induced charge is analyzed and applied again to the electrode 2, however, with an opposite sign to the induced charge and some phase advance. This causes a temporary halt of the oscillations that had induced such a charge.

The distance between the lever and the electrode 2 is chosen so that the electrical forces of the systems for indication of deflections and the for suppression of non-resonant oscillations would be significantly lower than the Van-der-Waals forces.

Example 7

The integration of several control systems in a single simple device, as it is proposed in this invention, allows to use such a device in the regime of multilever scanning of a highly-coarsed surface (see Fig. 23a). If use two or more levers for increasing the scanning rate, a problem arises: how to coordinate their operation if one of them should investigate a hollow whereas another one, spaced at some macroscopic distance from the former one, should investigate a hill? In the standard version of single lever, the problem is solved by feeding/removal of the holder. However, in the case of the multilever the problem can be solved by special techniques. In this invention, the feeding/removal of the lever by forced electrostatic deflection (E_2) is used.

In order to simplify the preparation of the multilever, a possibility to arranging several levers along the same electrode of the control systems is considered in this invention (see Fig. 23b).

In order to discriminate signals arrived to the systems for the indication of the deflection of the levers, it is sufficiently to apply signals of different frequencies to the pairs "lever-electrode 2".

For the system of the forced feeding/removal, as well as for the system of the suppression of non-resonant (parasitic) oscillations, it is necessary to decouple the levers each of other galvanically.

For a more precise investigation of the surface morphology, this invention considers a possibility to track the rotation modes around the elongated axis. To this aim, the lever is implemented in V- or Π- shaped version. If a single electrode 2 in Fig. 2 is arranged along each of arms of such a lever (see Fig. 23c), it is possible to obtain all the necessary information.

Example 8

A using multilayer composite wafer like silicon-on-insulator (SOI), where the lever is prepared from a silicon layer oriented along the plane (111), is the most typical method for preparation of the multilevers. Structures shown in Figs. 23a, b, c are prepared by repeated procedures of photolithography and etching.

After the structures have been prepared, whiskers were grown on them, and tips were prepared from the whiskers, as it was described above.

Terms and synonyms used in this invention:

Tip structure = substrate + tip(s);

Tip = links + steps;

Step = point of angle changing = point of changing any geometric parameter of the tip;

Branching point = step where the tip is doubling, thripling, etc.

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Claims

1. A tip structure that includes a single-crystalline substrate and a single-crystalline tip epitaxial to the substrate,

wherein

the axis of the tip forms a given angle in respect to the vertical that passes through its basis.

2. The tip structure according to the point 1, **wherein** the substrate has a plane surface.

3. The tip structure according to the point 1, **wherein** the substrate represents a single-crystalline tip epitaxial to a plane single-crystalline surface.

4. The tip structure according to any of the points 1-3, **wherein** a single point of the substrate serves as a basis for at least two tips.

5. The tip structure according to any of the points 1-4, **wherein** the tip has a shape that includes at least one step and two links, the axis of each subsequent link can form its own angle in respect to the axis of the previous link.

6. The tip structure according to the point 5, **wherein** at least one of the steps serves as a basis for at least two links, at least one of them can be not epitaxial to the previous one.

7. The tip structure according to any of the points 5 and 6, **wherein** at least one of the links is formed by a nanotube.

8. The tip structure according to the point 7, **wherein** the nanotube is combined by layers of different materials, one of them being carbon.

9. The tip structure according to any of the points 5-8, **wherein** at least one of the links is formed by at least one atomic row.

10. The tip structure according to any of the points 1-9, **wherein** at least one tip has a particle on its top that contains, in addition to the tip material, at least one more chemical element, the particle can be coated by a film of this or another element.

11. The tip structure according to the point 10, **wherein** at least one chemical element, that is contained in the particle, participates in the growing of the tip structure, and the particle can be coated by a film of this or another element.

12. The tip structure according to any of the points 10, 11, **wherein** chemical functional groups are deposited in the film.

13. The tip structure according to any of the points 1-8, **wherein** a non-magnetic tip has a flat top, a monodomenic magnetic particle of a conical shape is placed on the flat top, basis of the particle contacting with the flat top.

14. The tip structure according to any of the points 1-8, **wherein** an electroconductive tip has a flat top perpendicular to the axis of the tip, the flat top is coated by a dielectric film, a p-n junction in the upper part of the tip being parallel and close to the flat top.

15. A tip structure that includes a substrate and a single-crystalline tip

wherein

the tip is not epitaxial to the substrate.

16. The tip structure according to the point 15, **wherein** the axis of the tip forms an angle in respect to the vertical that passes through its basis.

17. The tip structure according to any of the points 15 and 16, **wherein** the substrate has a plane surface.

18. The tip structure according to any of the points 15-17, **wherein** a single-crystalline tip epitaxial to a plane single-crystalline surface serves as the substrate.

19. The tip structure according to any of the points 15 – 18, **wherein** one point of the substrate serves as a basis for at least two tips.

20. The tip structure according to any of the points 15-19, **wherein** the tip has a shape that contains at least one step and two links, the axis of each subsequent link has its own angle in respect to the axis of previous link.

21. The tip structure according to the point 20, **wherein** at least one step serves as a basis for two links, at least one of them can be not epitaxial to the previous one.

22. The tip structure according to the points 20 and 21, **wherein** at least one of the links is formed by a nanotube.

23. The tip structure according to the point 22, **wherein** the nanotube is combined of layers of different materials, one of them being carbon.

24. The tip structure according to any of the points 20-23, **wherein** at least one of the links is formed by at least one atomic row.

25. The tip structure according to any of the points 15-24, **wherein** at least one tip has a particle on its top that contains, in addition to the tip material, at least one more chemical element, the particle being coated by a film of this or another element.

26. The tip structure according to the point 25, **wherein** at least one chemical element, that is contained in the particle, participates in the growing of the tip structure, and the particle can be coated by a film of this another element.

27. The tip structure according to any of the points 25, 26, **wherein** chemical functional groups are deposited on the film.

28. The tip structure according to any of the points 15-24, **wherein** a non-magnetic tip has a flat top, a monodomenic magnetic particle of a conical shape is placed on the flat top, basis of the particle contacting with the flat top.

29. The tip structure according to any of the points 15-24, **wherein** an electroconductive tip has a flat top perpendicular to its axis, the flat top is coated by a dielectric film, a p-n junction in the upper part of the tip being parallel and close to the flat top.

30. A method for preparation of the tip structure by means of epitaxial growing of the tip according to the vapor-liquid-solid mechanism on a substrate by deposition from a vapor-gaseous and/or gaseous mixture with using of at least one metallic solvent,

wherein

the tip structure is growing as at least one tip so that the axis of the tip forms a given angle in respect to the vertical that passes through its basis.

31. The method according to the point 30, **wherein** as the substrate a single-crystalline wafer oriented along a certain crystallographic plane is used, this single-crystalline wafer allows to prepare the tip structure as at least one tip epitaxial to the substrate under an angle to its surface.

32. The method according to the point 30, **wherein** a single-crystalline tip epitaxial to a flat single-crystalline surface is used as the substrate.

33. The method according to any of the points 30-32, **wherein** the tip structure of the points 3-14 is prepared by a changing the growing temperature and/or concentrations of compounds in the vapor-gaseous or gaseous mixture, and/or

pressures of the vapor-gaseous or gaseous mixture, and/or by addition of at least one metallic solvent and/or its evaporation.

34. The method according to any of the points 30-33, wherein after the growing the tip structure a diffusion of at least one chemical element into the structure is performed with conservation of the structure of at least one metallic solvent.

35. The method according to any of the points 30-34, **wherein** after the preparation of the structure it is immersed into an amorphous material, the composite obtained is polished together with at least one apex of the tip structure until formation of a flat surface, and the amorphous material can be etched away.

36. The method according to any of the points 30-35, **wherein**, after the etching away the amorphous material, a diffusion of at least one chemical element into the material of the tip structure is performed.

37. The method according to any of the points 30-36, wherein a diffusion of at least one chemical element into at least one metallic solvent is performed.

38. The method according to the point 37, **wherein** by etching off the material that has diffused into at least one metallic solvent, the metallic solvent is removed.

39. The method according to any of the points 30-38, **wherein** at least one chemical element is evaporated onto all the surface of the tip structure.

40. The method according to the point 39, **wherein** a part of the evaporated chemical element is removed by an etching off the diffusion layer from the surface of the tip structure, or by etching off the amorphous layer with conservation of the chemical element on at least one apex.

41. The method according to any of the points 30-40, **wherein** the epitaxial growing of the tip structure is implemented, and by a changing the growing temperature and/or concentrations of compounds in the vapor-gaseous or gaseous mixture, and/or pressures of the vapor-gaseous or gaseous mixture, and/or by addition of at least one metallic solvent and/or its evaporation a step and/or a plateau on at least one apex is created, after that the solidified globule can be removed.

42. The method according to any of the points 34-41, **wherein** at least one of the procedures described in the points 33-41 is used at least one more time.

43. The method according to any of the points 39-41, **wherein** at least one time a magnetic material is used as the evaporating one, the magnetic particle formed

is sharpened by a bombardment with accelerated ions, and a monodomenization of the particle can be performed.

44. The method according to the point 43, **wherein** the monodomenization is performed by enduring of the particle in a constant magnetic field of a certain direction.

45. The method according to the point 43 and 44, **wherein** the monodomenization is performed at a high temperature of the magnetic particle, the temperature can be reached by passing a field-emission current through the tip structure.

46. A method for preparation of at least one tip structure by directional growing according to the vapor-liquid-solid mechanism on a substrate at deposition from a vapor-gaseous and/or gaseous mixture with using of at least one metallic solvent,

wherein

the tip structure is grown non-epitaxially to the substrate.

47. The method according to the point 46, **wherein** the tip structure is created according to the points 15-29.

48. The method according to any of the points 46 and 47, wherein a hollow is created in the substrate for the growing of the tip

49. The method according to the point 48, **wherein** the hollow has a shape that corresponds to the crystallographic structure of the tip material.

50. A source of electrons that includes a substrate, a field emitter, and a source of charge carriers,

wherein

the field emitter represents a tip structure according to the points 1-11 and 15-26.

51. A cantilever for scanning probe devices that includes a holder, a lever and a probe,

wherein

the tip is implemented as a tip structure according to the points 1-29.

52. A cantilever for scanning probe devices that includes:

at least two alternating plane-parallel layers of conducting materials separated by non-conducting layers;

at least one bending section – a lever implemented from a first conducting layer;

a probe placed on the lever;

at least one electrode – a section of a second conducting layer arranged along the lever at the side opposite to the probe;

wherein

the electrode contains a means for suppression of non-resonant oscillations of the lever, the means representing a feedback system.

53. The cantilever according to the point 52, **wherein** the probe is implemented as a tip structure according to the points 1-29.

54. The cantilever according to any of the points 52 and 53, **wherein** the electrode contains a means for controlling the lever deflections, and/or a means for a forced deflection of the lever of its initial position, and/or a means for modulation of the resonant lever oscillations.

55. The cantilever according to any of the points 52-54, **wherein** at the side of the lever that is opposite to the electrode side of the lever is arranged another electrode implemented from an additional conducting layer and contained a means for controlling lever deflections, and/or a means for a forced lever deflection of its initial position, and/or a means for modulations of resonant oscillations of the lever, and/or a means for suppression of non-resonant oscillations of the lever, the system being acted as a feedback one.

56. The cantilever according to any of the points 52-55, **wherein** between the lever and at least one electrode a vacuum gap exists, the gap can be filled by liquid and/or plastic material that allows a mutual shifting of the lever and the electrode relative to each other.

57. The cantilever according to any of the points 52-56, **wherein** the lever has a Π- and/or V-shape and/or longitudinal cavity, the cavity forming lever arms.

58. The cantilever according to any of the points 52-57, **wherein** the lever has a piezoresistive layer and/or semiconductor layer doped up to the p^{++}-conductivity.

59. The cantilever according to any of the points 57 and 58, **wherein** the lever arms separated by a longitudinal section has doped layers of n-, n^+-, p-, p^+ type conductivity.

60. The cantilever according to any of the points 57 and 59, **wherein** one of the lever arm serves as a drain, another lever arm serves as a source for a control system, the arms being separated by a lever section that has another conductivity, one of the electrodes implements a function of a gate being a means of a control.

<u>**61**</u>. A scanning probe device that includes:

a cantilever containing at least one lever, at least one controlling electrode and/or at least one electrode for controlling lever deflections of an initial position;

a system for regulation and controlling the lever deflections

<u>**wherein**</u>

the lever is implemented according to any of the points 52-60.

62. The scanning probe device according to the point 61, **wherein** one electrode is placed along at least two levers.

63. The scanning probe device according to any of the points 61 and 62, **wherein** the system for controlling the lever deflections represents a system for registration of the changes of the capacity between the lever and at least one electrode and/or of the contour quality, that includes its capacity, for each lever being chosen its own control frequency.

64. The scanning probe device according to any of the points 61-63, **wherein** the control system includes a system for the forced deflection that can be electrostatic and/or electromagnetic one.

65. The scanning probe device according to any of the points 61, 63, and 64, **wherein** at least two electrodes are placed along the same lever.

66. The scanning probe device according to the point 65, **wherein** the system for controlling the lever deflections represents a system for registration of the changes of the capacity between the arms of the lever, separated by a longitudinal section, and at least one electrode, for a controlling the lever rotation relative to a longitudinal axis a specific frequency being chosen for each of the lever arms.

<u>**67**</u>. A method for preparation of a cantilever for scanning probe devices that includes

a formation of a composite wafer consisting of at least two alternating plane-parallel layers of conducting materials separated by non-conducting layers;

a formation of at least one lever from the first conducting layer;

a creation of a probe on the lever

wherein

at least one electrode arranged along the lever at a side opposite to the probe is formed from the second conducting layer.

68. The method according to according to the point 67, **wherein** the probe is implemented as the tip structure according to the points 1-14 and 15-29.

69. The method according to any of the points 67 and 68, **wherein** the composite wafer is prepared by bonding of wafers and/or mechanical and/or chemical removal of parts of the wafers with conservation of thin layers having a given thickness.

70. The method according to any of the points 67-69, **wherein** at least one conducting layer and/or at least one non-conducting layer of the composite wafer are prepared by a deposition of a material and/or materials.

71. The method according to any of the points 67-70, **wherein** an electrode with contact outputs/terminals and/or a mechanico-electric structure for systems of control and/or regulation is formed on at least one conducting layer before the bonding and/or between bonding stages and/or after the bonding.

72. The method according to any of the points 71 and 72, **wherein** an electrode with contact outputs/terminals and/or a mechanico-electric structure for systems of control and/or regulation is formed on at least one conducting layer before the deposition and/or between the deposition stages and/or after the deposition of the material and/or materials.

73. The method according to any of the points 67-72, **wherein** at least one conducting layer and/or at least one non-conducting layer are used at the preparation of the cantilever as technological stop-layers.

74. The method according to any of the points 67-73, **wherein** the probe is implemented as the tip structure according to points 30-49.

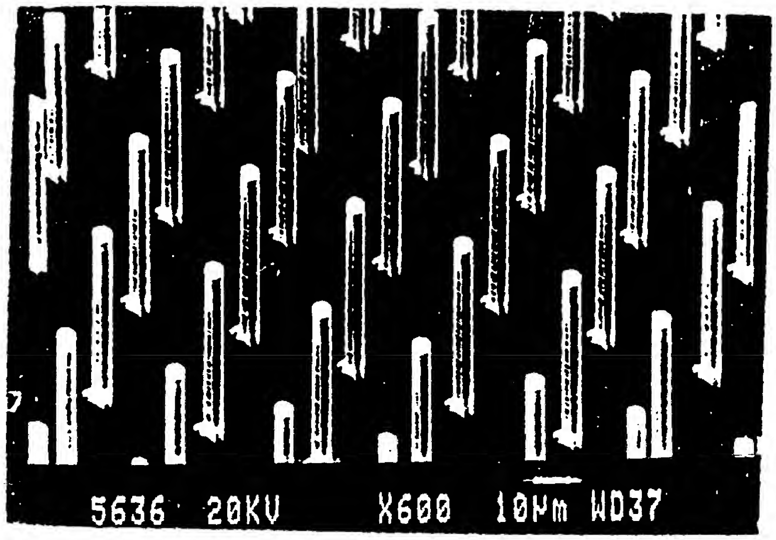
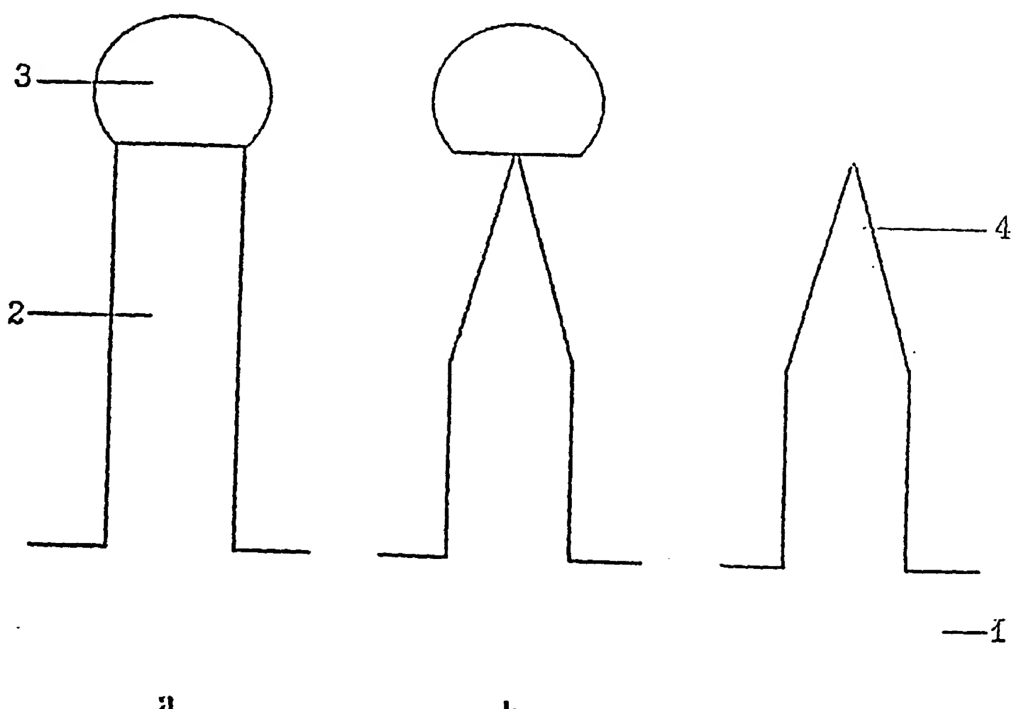


Fig. 1.



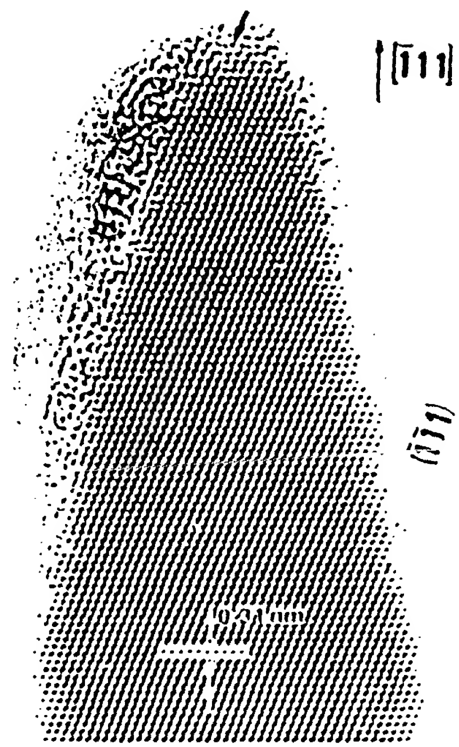


Fig. 3.

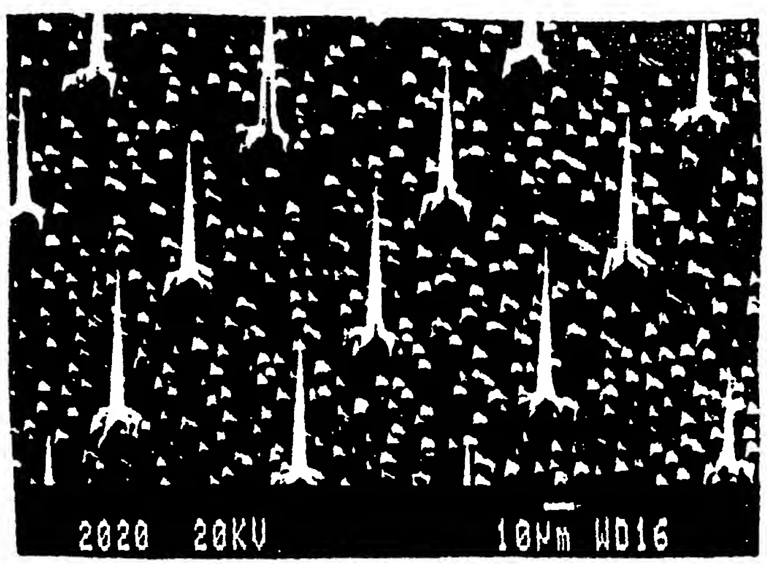
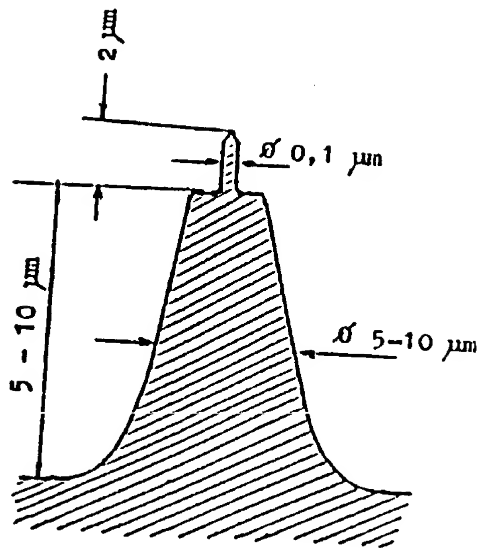


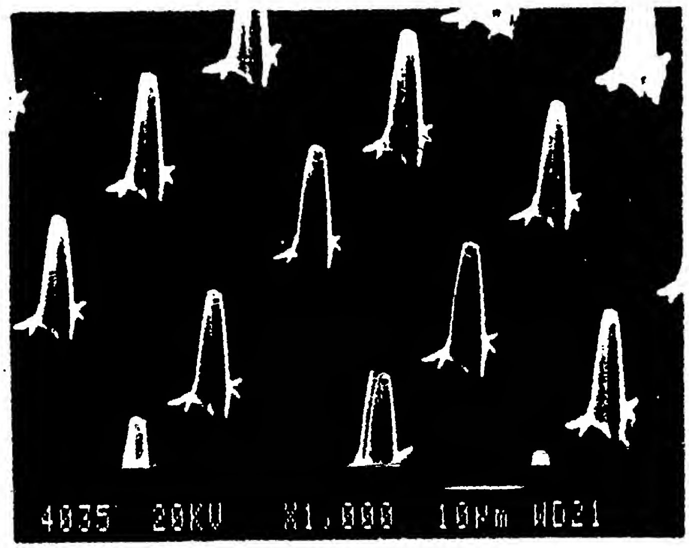
Fig. 4.

a



b

Fig. 5.



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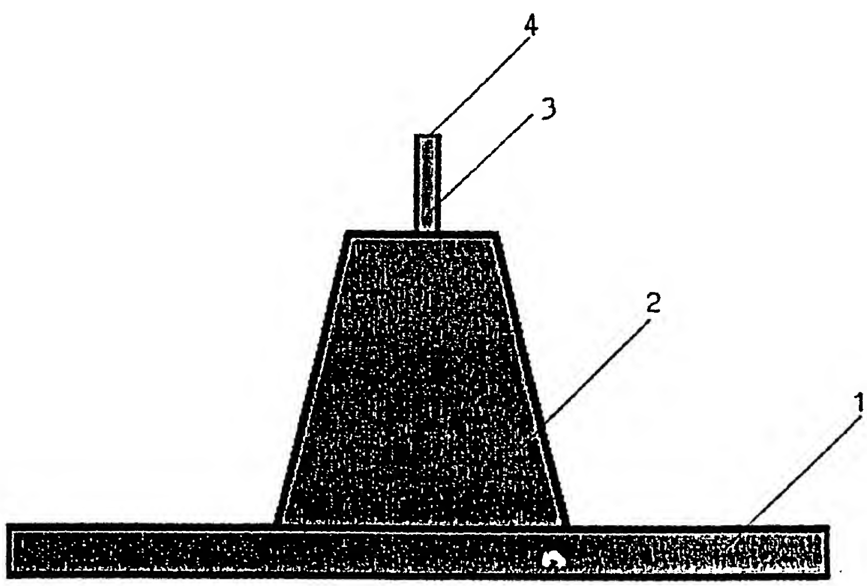
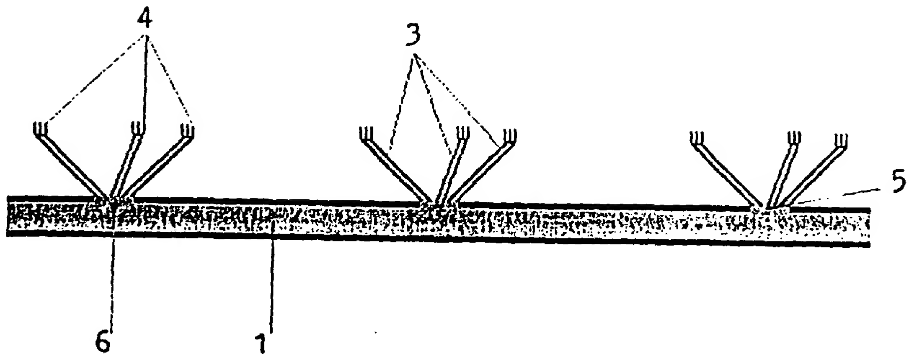


Fig. 7.

a



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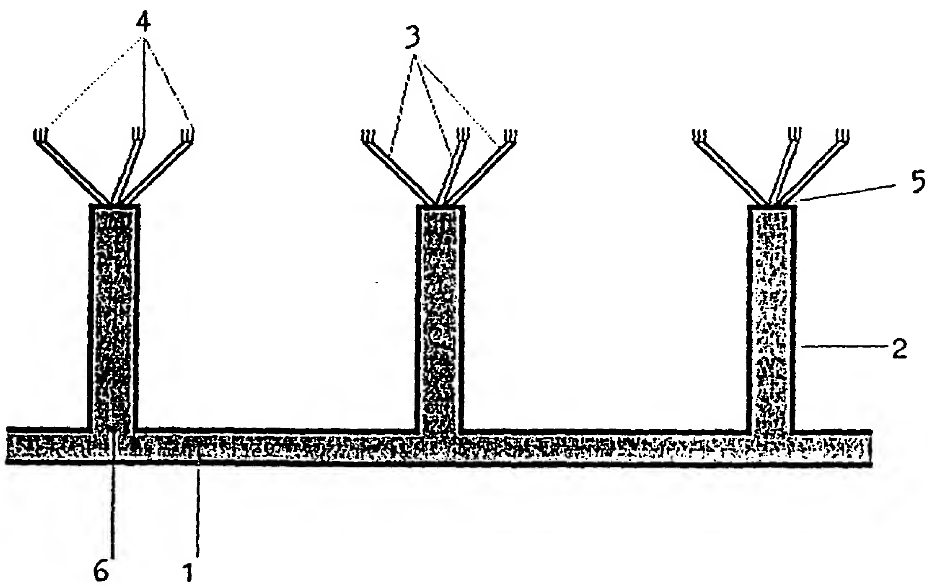


Fig. 8.

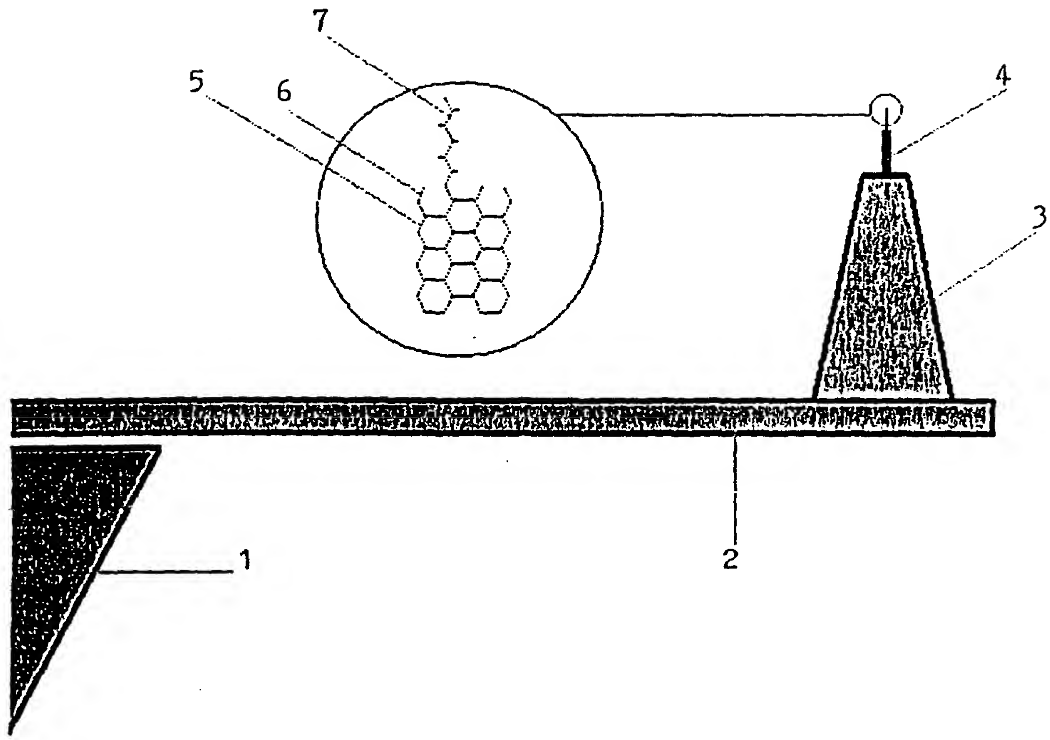


Fig. 9.

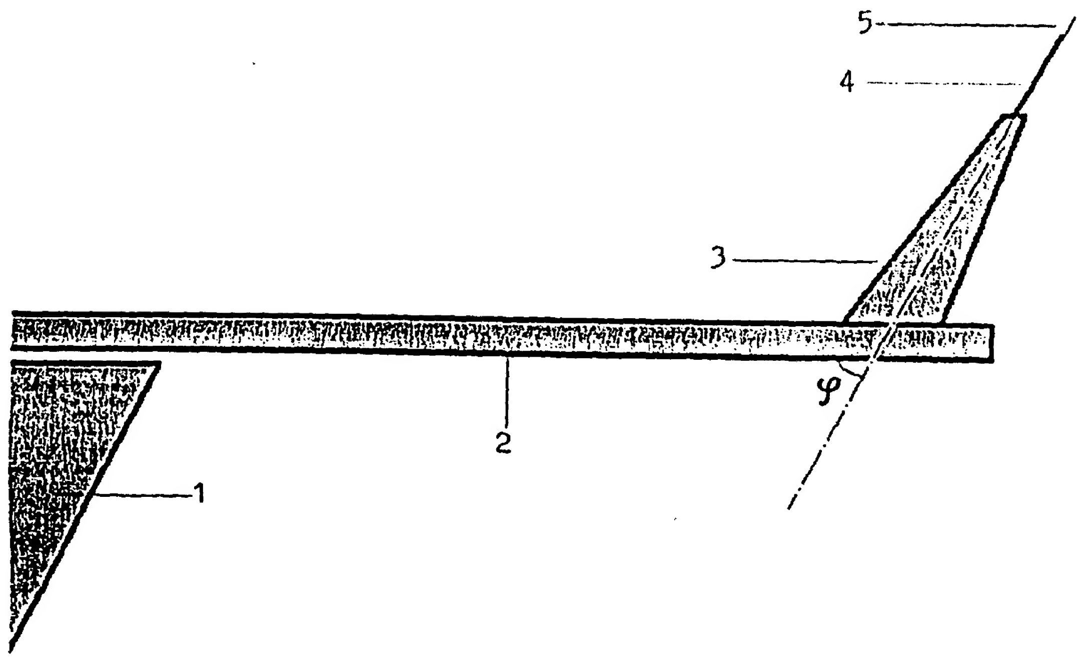


Fig. 10

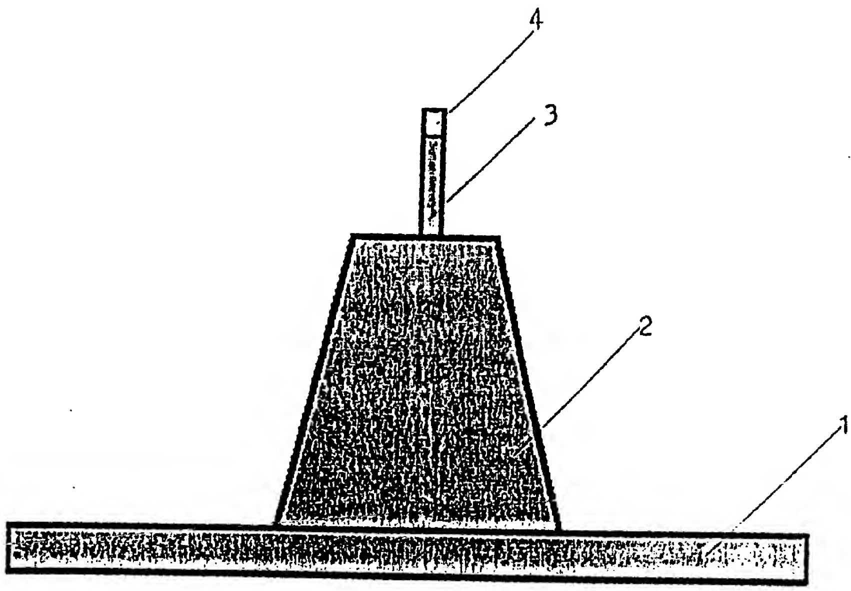


Fig. 11.

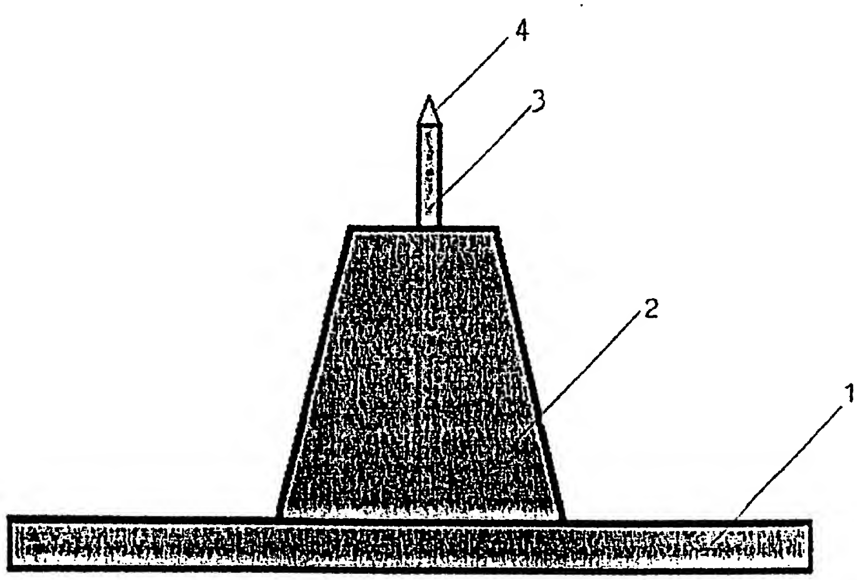
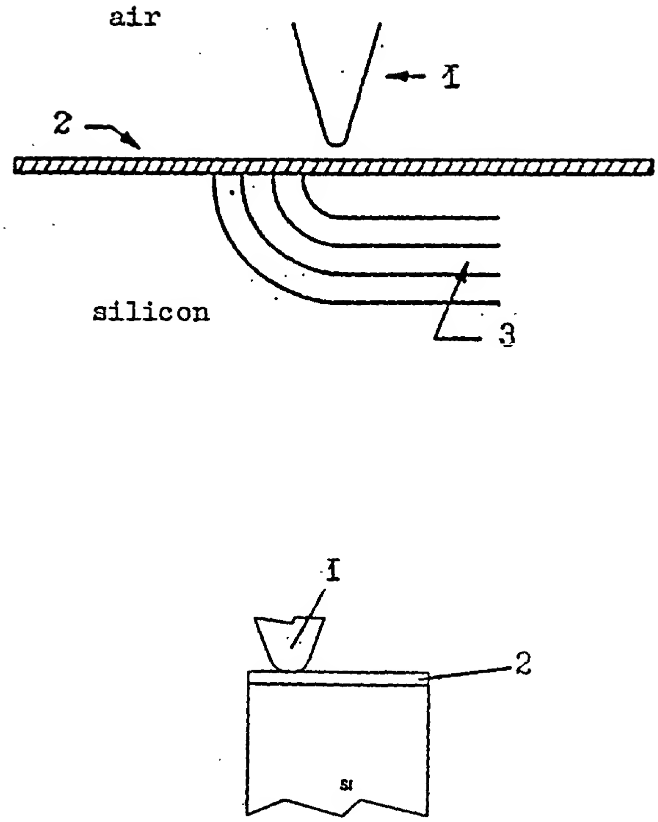


Fig. 12.



a

b

Fig. 13.

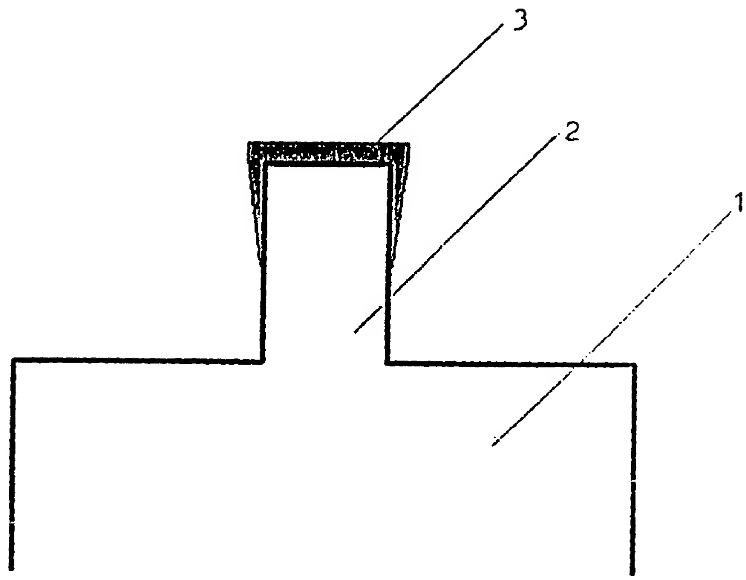


Fig. 14.

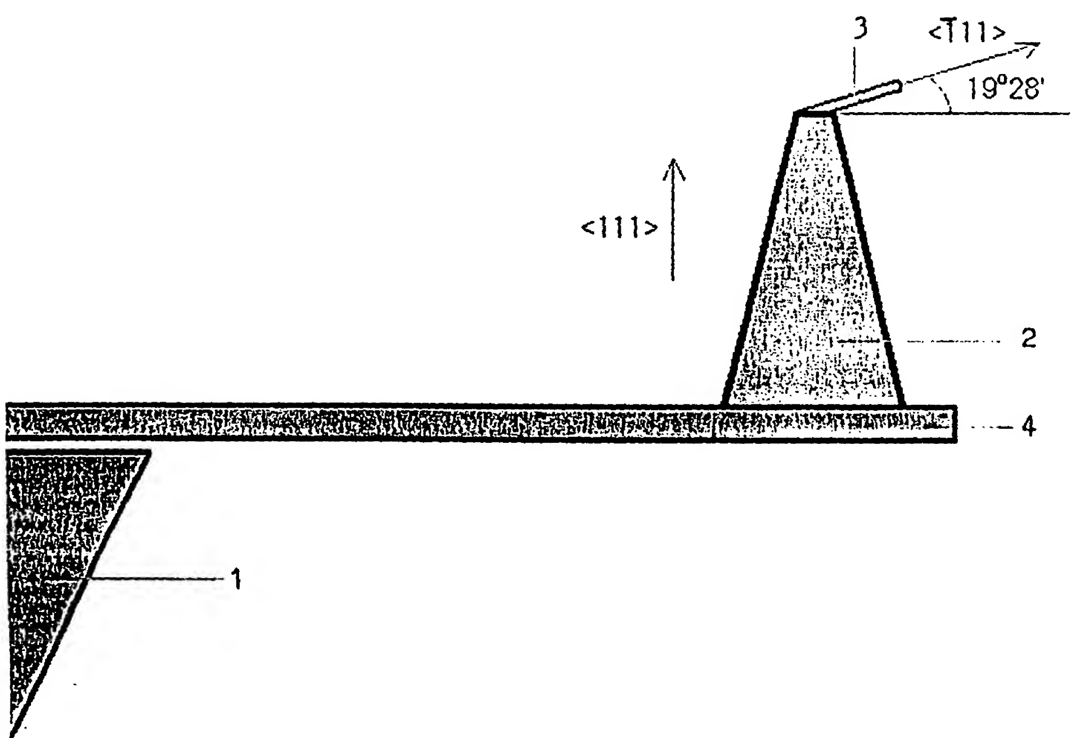


Fig. 15.

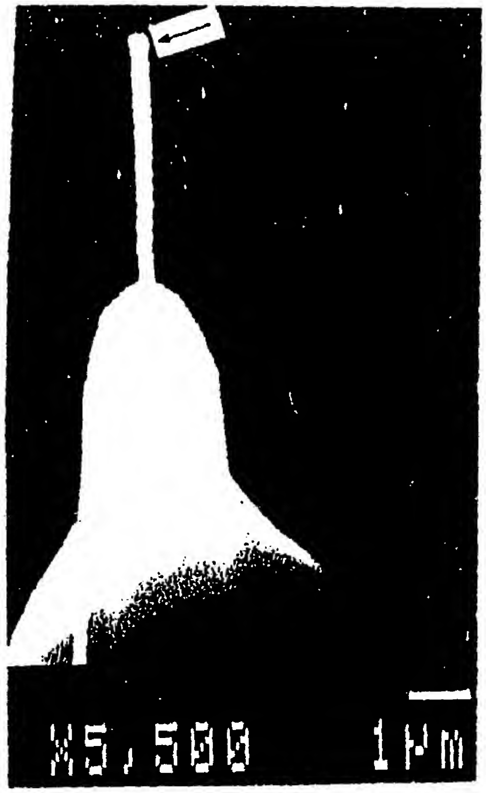


Fig. 16.

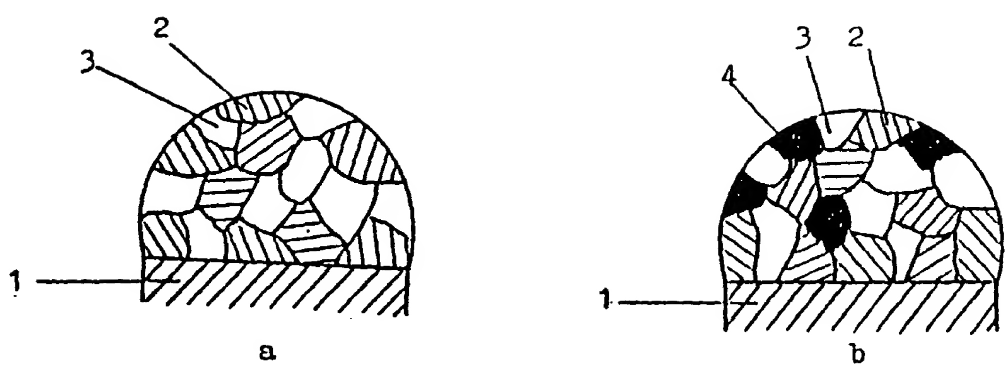


Fig. 17.

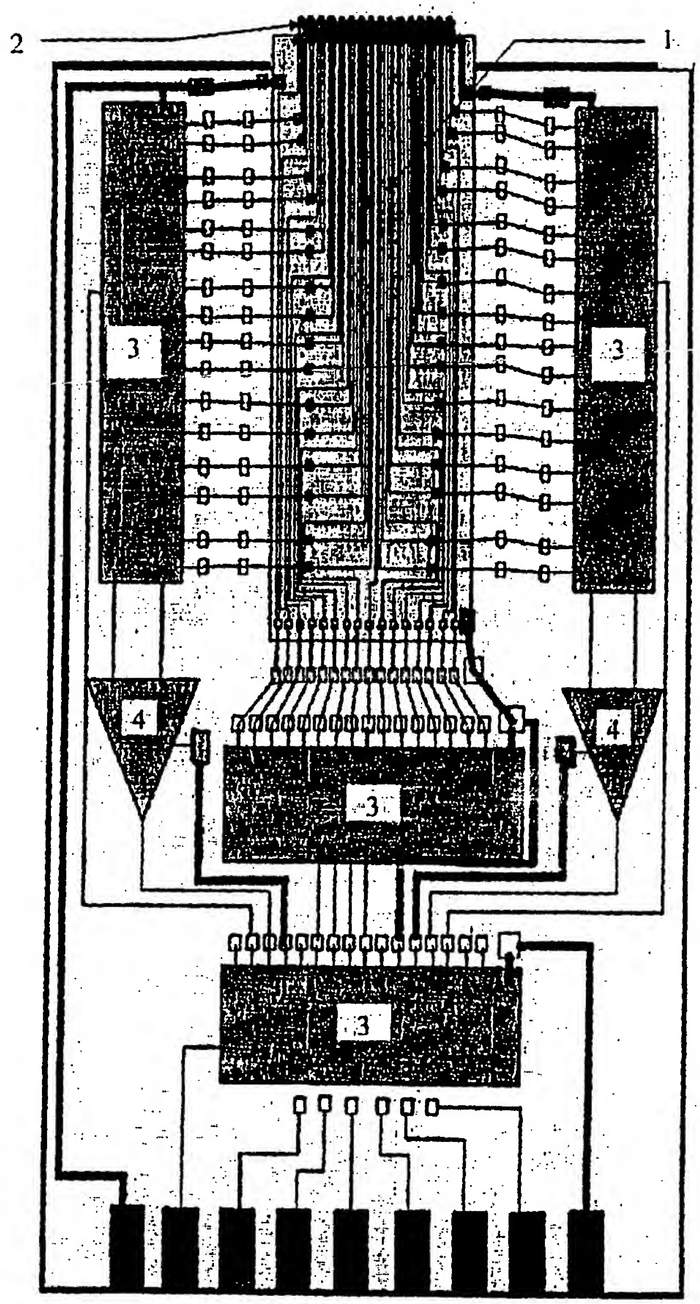


Fig. 18

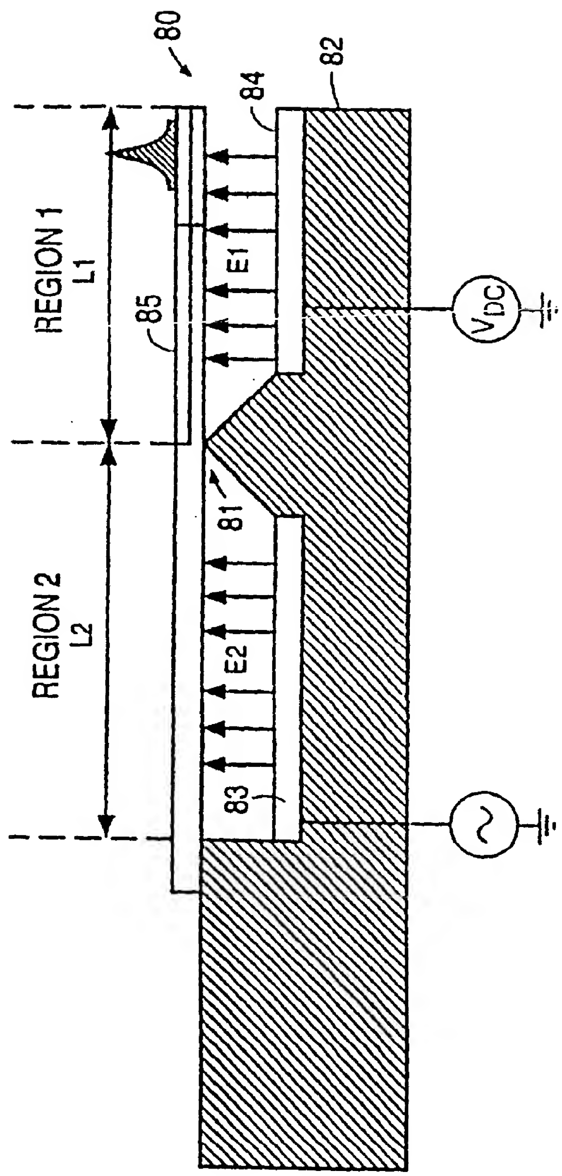


Fig. 19

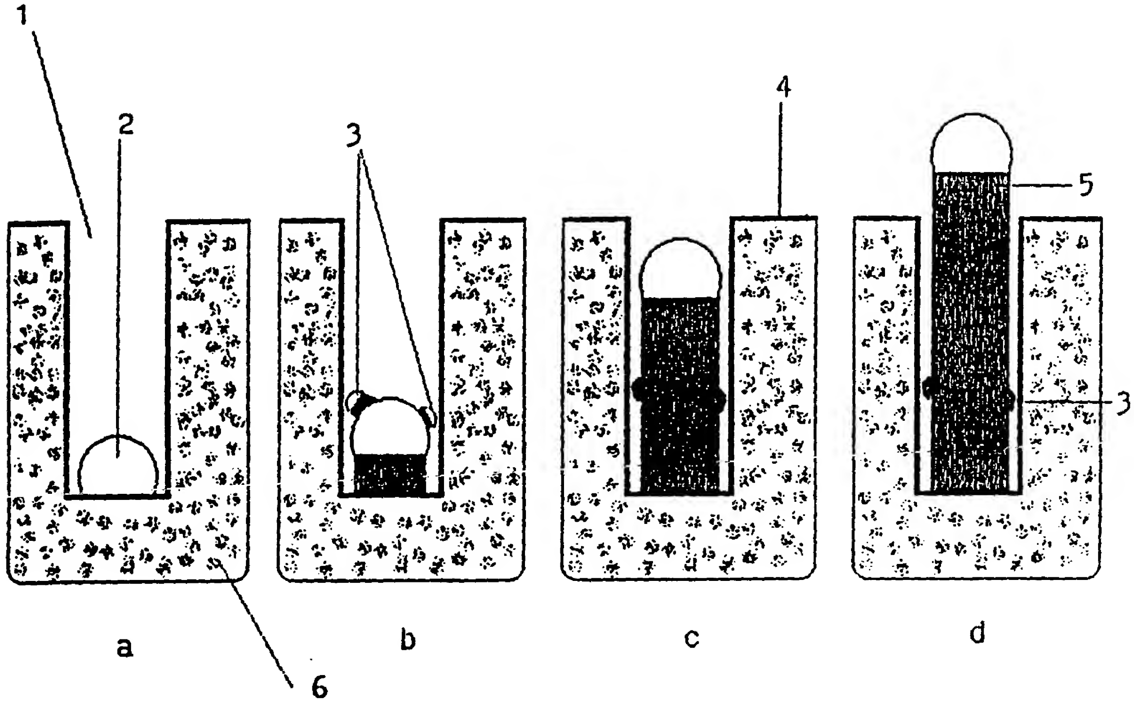


Fig. 20

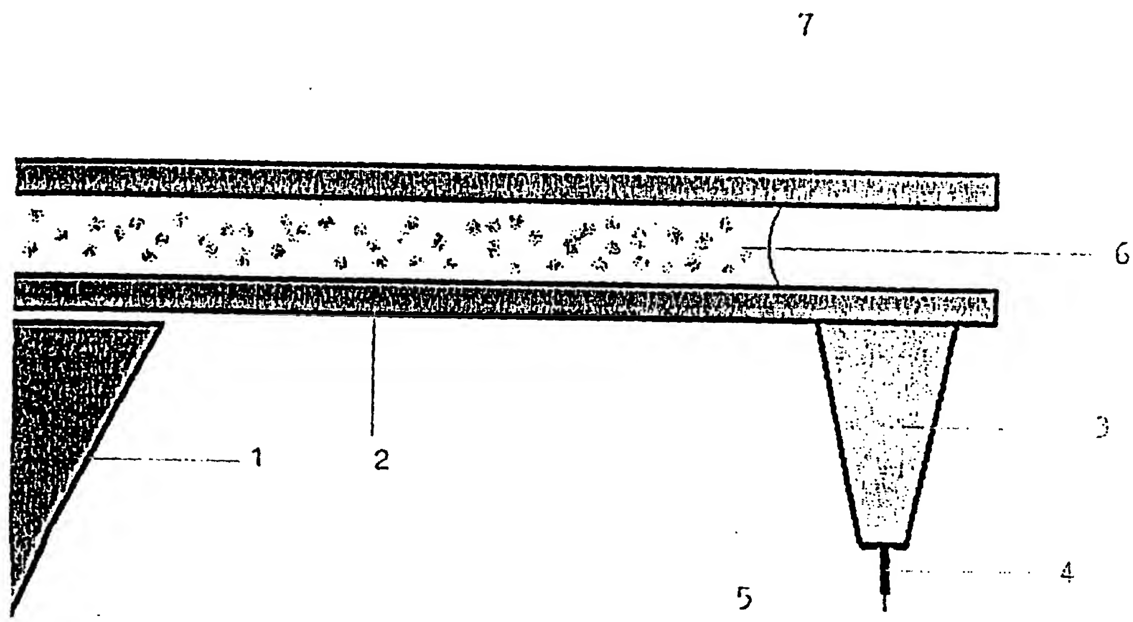


Fig. 21

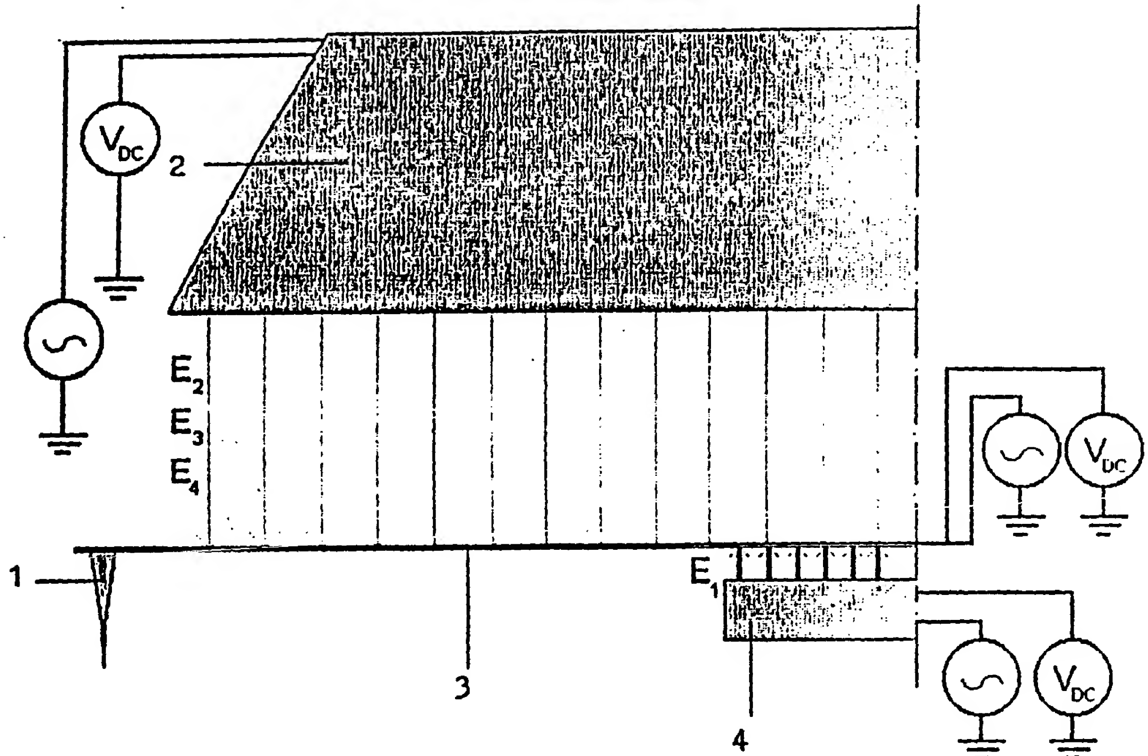


Fig. 22

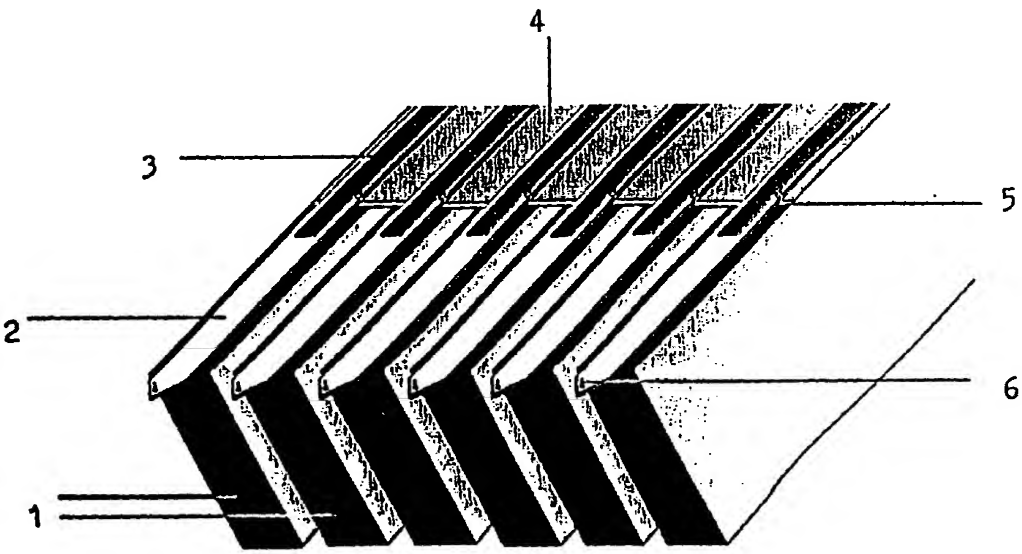


Fig. 23 a

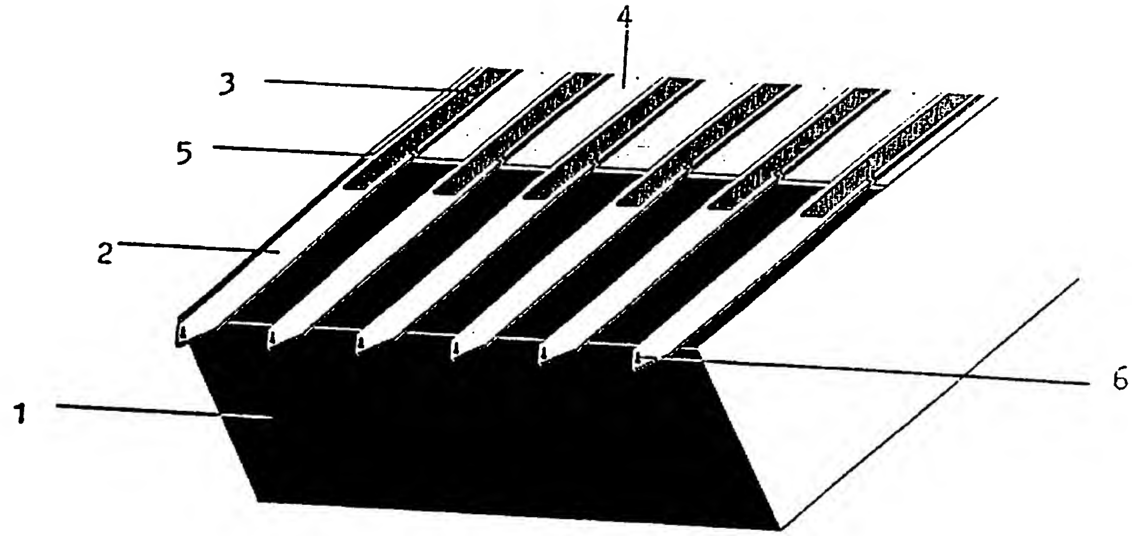
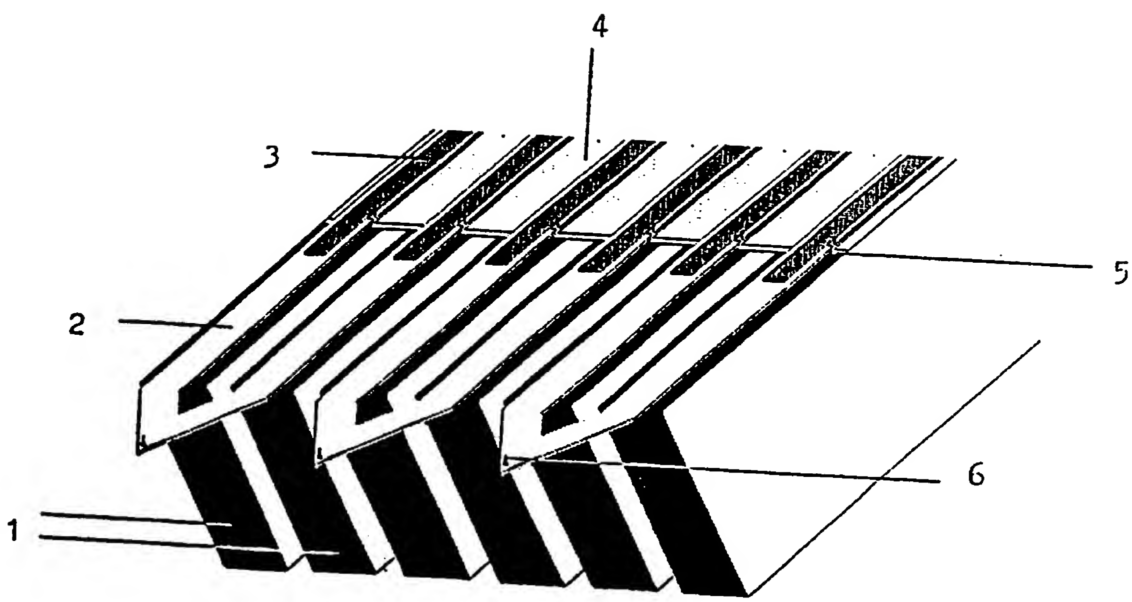


Fig. 23 b



(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
7 December 2000 (07.12.2000)

PCT

(10) International Publication Number
WO 00/74107 A2

(51) International Patent Classification[7]: H01L

(21) International Application Number: PCT/RU00/00209

(22) International Filing Date: 31 May 2000 (31.05.2000)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:

99111382	31 May 1999 (31.05.1999)	RU
99111432	31 May 1999 (31.05.1999)	RU
2000107025	23 March 2000 (23.03.2000)	RU
2000107026	23 March 2000 (23.03.2000)	RU

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(81) **Designated States** *(national)*: AT, AU, BG, BR, CA, CH, CN, CZ, DE, DK, ES, FI, GB, HU, IL, IN, JP, KR, LT, LU, PL, RU, SE, SG, SI, SK, UA, US.

(84) **Designated States** *(regional)*: European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

Published:

— *Without international search report and to be republished upon receipt of that report.*

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WO 00/74107 A2

(54) **Title:** TIP STRUCTURES, DEVICES ON THEIR BASIS, AND METHODS FOR THEIR PREPARATION

(57) **Abstract:** New designs of electron devices such as scanning probes and field emitters based on tip structures are proposed. The tips are prepared from whiskers that are grown from the vapor phase by the vapor-liquid-solid technology. Some new designs for preparation of field-emitters and of probes for magnetic, electrostatic, morphological, etc, investigations based on the specific technology are proposed. New designs for preparation of multilever probes are proposed, too.

PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference 10G	**FOR FURTHER ACTION** see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. PCT/RU 00/ 00209	International filing date *(day/month/year)* 31/05/2000	(Earliest) Priority Date *(day/month/year)* 31/05/1999
Applicant Givargisov Evgeny Invievich		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 4 sheets.

[X] it is also accompanied by a copy of each prior art document cited in this report.

1. **Basis of the report**

   a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

   [ ] the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

   b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :

   [ ] contained in the international application in written form.
   [ ] filed together with the international application in computer readable form.
   [ ] furnished subsequently to this Authority in written form.
   [ ] furnished subsequently to this Authority in computer readble form.
   [ ] the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
   [ ] the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. [ ] **Certain claims were found unsearchable** (See Box I).
3. [ ] **Unity of invention is lacking** (see Box II).

4. With regard to the **title,**

   [X] the text is approved as submitted by the applicant.
   [ ] the text has been established by this Authority to read as follows:

5. With regard to the **abstract,**

   [ ] the text is approved as submitted by the applicant.
   [X] the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No. 10

   [ ] as suggested by the applicant.
   [X] because the applicant failed to suggest a figure.
   [ ] because this figure better characterizes the invention.
   [ ] None of the figures.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/RU 00/00209

Box III TEXT OF THE ABSTRACT (Continuation of item 5 of the first sheet)

New designs of electron devices such as scanning probes and field emitters based on tip structure are proposed. The tips are prepared from whiskers that are grown from the vapor phase by the vapo-liquid-solid technology. The tip structure includes a single crystalline substrate and a single crystalline tip. The axes of the tip forms a given angle in respect to the vertical that passes through its basis.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/RU 00/00209

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 G01B7/34 G01N27/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 G01B G01N G01R C30B H01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

PAJ, WPI Data, INSPEC, COMPENDEX, EPO-Internal, IBM-TDB

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 367 165 A (TODA AKITOSHI ET AL) 22 November 1994 (1994-11-22) claims 1-9	1,51,52
A	US 5 742 377 A (MINNE STEPHEN CHARLES ET AL) 21 April 1998 (1998-04-21) cited in the application claims 1-38	51,52,61
A	WO 97 37064 A (GIVARGIZOV EVGENY INVIEVICH) 9 October 1997 (1997-10-09) claim 1	30,46
A	US 5 811 017 A (MATSUYAMA KATSUHIRO) 22 September 1998 (1998-09-22) claim 1	1,30

-/--

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
29 November 2000	18/12/2000

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Van den Bulcke, E

2

Form PCT/ISA/210 (second sheet) (July 1992)

In onal Application No
PCT/RU 00/00209

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 021 364 A (AKAMINE SHINYA ET AL) 4 June 1991 (1991-06-04) claims 1-16	1,30
A	US 5 903 161 A (NAKANO TATSUO ET AL) 11 May 1999 (1999-05-11) claims 1,10,11	1
A	EP 0 566 214 A (CANON KK) 20 October 1993 (1993-10-20) claims 1-4	1
A	A.A.NOSOV ET AL.: "the influence of an electric field on the formation of gold whisker single crystals" RADIO ENGINEERING AND ELECTRONIC PHYSICS, vol. 17, no. 2, February 1972 (1972-02), pages 281-286, XP002154139 page 281 -page 286; figures 2-7	1
A	K.L.LEE ET AL.: "SUBMICRON Si TRENCH profiling with an electron -beam fabricated atomic force microscope tip" JOURNAL VACUUM SCIENCE TECHNOLOGY , vol. B9, no. 6, November 1991 (1991-11), pages 3562-3568, XP000965676 cited in the application page 3564; figure 3B	1
A	E.I.GIVARGIZOV: "ultrasharp tips for field emission applications prepared by the vapor-liquid-solid growth technique" JOURNAL VACUUM SCIENCE TECHNOLOGY, vol. b, no. 11(2), March 1993 (1993-03), pages 449-453, XP000965673 cited in the application page 449; figure 453	

2

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/RU 00/00209

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5367165 A	22-11-1994	JP 6052820 A	25-02-1994
		JP 5256643 A	05-10-1993
US 5742377 A	21-04-1998	US 5517280 A	14-05-1996
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		JP 2553661 B	13-11-1996

PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant s or agent s file reference 10G	**FOR FURTHER ACTION**	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
International application No. PCT/RU 00/00209	International filing date (day/month/year) 31 May 2000 (31.05.2000)	Priority date (day/month/year) 31 May 1999 (31.05.1999)
International Patent Classification (IPC) or national classification and IPC		G01B 7/34, G01N 27/00
Applicant	GIVARGIZOV Evgeny Invievich et al.	

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This Report consists of a total of 3 sheets, including this cover sheet.

   ☐ This report is also accompanied by ANNEXES, i.e., sheets of the description, claimes and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under PCT).

   These annexes consist of a total of ______ sheets

3. This report contains indications relating to the following items:

   I ☒ Basis of the report

   II ☐ Priority

   III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability

   IV ☐ Lack of unity of invention

   V ☒ Reasoned statement under Articl 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

   VI ☐ Certain documents cited

   VII ☐ Certain defects in the international application

   VIII ☐ Certain observations on the international application

Date of submission of the demand: 29 December 2000 (29.12.2000)	Date of completion of this report: 05 August 2001 (05.08.2001)
Name and mailing address of the IPEA/ RU FIPS Russia, 121858, Moskva, Berezhkovskaya nab., 30-1 Facsimile No.	Authorized officer E.Andreichenko Telephone No 240-25-91

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/RU 00/00209

I. Basis of the report

1. With regard to the **elements** of the international application:*

[X] the international application as originally filed

[ ] the description:
pages ______, as originally filed
pages ______, filed with the demand
pages ______, filed with the letter of ______

[ ] the claims:
pages ______, as originally filed
pages ______, as amended (together with statement) under Article 19
pages ______, filed with the demand
pages ______, filed with the letter of ______

[ ] the drawings:
pages ______, as originally filed
pages ______, filed with the demand
pages ______, filed with the letter of ______

[ ] the sequence listing part of the description:
pages ______, as originally filed
pages ______, filed with the demand
pages ______, filed with the letter of ______

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.
These elements were available or furnished to this Authority in the following language ______ which is:

[ ] the language of a translation furnished for the purposes of international search (under Rule 23.1.(b)).

[ ] the language of publication of the international application (under Rule 48.3(b)).

[ ] the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

[ ] contained in the international application in written form.

[ ] filed together with international application in computer readable form.

[ ] furnished subsequently to this Authority in written form.

[ ] furnished subsequently to this Authority in computer readable form.

[ ] The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

[ ] The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. [ ] The amendments have resulted in the cancellation of:

[ ] the description, pages ______
[ ] the claims, Nos. ______
[ ] the drawings, sheets/fig. ______

5. [ ] This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).**

* *Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).*

** *Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.*

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/RU 00/00209

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicabillity; citations and explanations supporting such statement

1. Statement

Novelty (N)	Claims	1 - 74	YES
	Claims		NO
Inventive Step (IS)	Claims	1 - 74	YES
	Claims		NO
Industrial Applicability (IA)	Claims	1 - 74	YES
	Claims		NO

2. Citations and explanations (Rule 70.7)

The examination report has been established on the basis of all the documents cited in Search report. The following documents are considered to be the closest prior art for the claimed inventions:

D1 - US 5367165 D3 - WO 97/37064

D2 - US 5742377 D4 - US 5811017

D1, D3, D4 disclose the tip structures, the methods for preparation of the tip structures and the cantilevers including the tip structures. D2 discloses the cantilever, the method for its preparation and the scanning probe device including the cantilever. But none of these documents discloses the features of claim 1 and 30, namely the axis of the tip forming a given angle in respect to the vertical that passes through its basis. None of these documents discloses the features of independent claims 15 and 46 concerning the tip not epitaxial to the substrate. None of the documents discloses the features of claim 51 concerning the tip implemented as a tip structure according to claims 1-29. The invention according to claim 67 differs from known by that a method for preparation of a cantilever includes a formation of at least one lever from the first conducting layer and a formation of at least one electrode arranged along the lever at a side opposite to the probe from the second conducting layer.

The claimed inventions provide the possibility of creating new designs of electron devices such as scanning probes and field emitters based on tip structures.

Consequently claims 1-74 are novel and involve an inventive step.